PB-245 050

DEVELOPMENT OF TECHNICAL SPECIFICATIONS FOR STANDARD GAS-DILUENT MIXTURES FOR USE IN MEASUREMENT OF MOBILE SOURCE EMISSIONS

Louis R. Reckner

Scott Research Laboratories,
Incorporated

Prepared for:

National Environmental Research Center

June 1974

DISTRIBUTED BY:

NTIS

National Technical Information Service
U. S. DEPARTMENT OF COMMERCE

272228

PB 245 050

EPA-650/4-74/020

June 1974

Environmental Monitoring Series

DEVELOPMENT OF TECHNICAL SPECIFICATIONS FOR STANDARD GAS-DILUENT MIXTURES FOR USE IN MEASUREMENT OF MOBILE SOURCE EMISSIONS

Reproduced by
NATIONAL TECHNICAL
INFORMATION SERVICE
US Department of Commerce
Springfield, VA. 22151

Office of Research and Development
U.S. Environmental Protection Agency
Washington, DC 20460

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO.	2	3. RECIPIENT'S ACCESSION NO.
EPA 650/4-74-020		

4. TITLE AND SUBTITLE	5. REPORT DATE
Development of Technical Specifications For Standard Gas-Diluent Mixtures for Use in Measurement of Mobile Source Emissions	June 1974
	6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) Louis R. Reckner	8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS Scott Research Laboratories, Inc. Plumsteadville, Pa. 18949	10. PROGRAM ELEMENT NO. 1HA327
	11. CONTRACT/GRANT NO. 68-02-0652
12. SPONSORING AGENCY NAME AND ADDRESS Office of Research and Development U.S. Environmental Protection Agency Washington, D.C. 20460	13. TYPE OF REPORT AND PERIOD COVERED
	14. SPONSORING AGENCY CODE

15. SUPPLEMENTARY NOTES

16. ABSTRACT

The stability of gas mixtures of carbon monoxide in nitrogen and propane in air in high-pressure cylinders was investigated in 240 test cylinders over a six month period. The effect of several variables related to the preparation of the mixtures by the suppliers, the storage of the cylinders and their use by laboratories engaged in emission measurements was studied. The variables included:

1. Cylinder wall material; 2. Cylinder valve type; 3. Cylinder preconditioning procedure; 4. Concentration of carbon monoxide and propane; 5. Purity of diluent nitrogen and air,; 6. Mixing procedure after blending; 7. Temperature at which cylinders are stored; 8. Cylinder pressure; 9. Type of pressure-reducing regulator used.

The concentration data obtained by periodic analysis of the 240 cylinders over the six month period were subjected to statistical analysis by multiple stepwise regression. The effects of the individual variables are discussed, and recommended practices for assuring stable mixtures of carbon monoxide in nitrogen and propane in air are presented.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS		b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Air Pollution	Nitrogen	Standard Gas-Diluent mixtures	13B
Carbon Monoxide	Air		7B
Propane	Tests		7C
Stability		Mobile Source Emissions	
Gas Cylinders	Specifications		

18. DISTRIBUTION STATEMENT	19. SECURITY CLASS (This Report)	21. NO. OF PAGES
Unlimited	Unclassified	65
	20. SECURITY CLASS (This page) Unclassified	22. PRICE 4.25

EPA Form 2220-1 (9-73)

EPA-650/4-74/020

DEVELOPMENT OF TECHNICAL SPECIFICATIONS FOR STANDARD GAS-DILUENT MIXTURES FOR USE IN MEASUREMENT OF MOBILE SOURCE EMISSIONS

by

Louis R. Reckner

Scott Research Laboratories, Inc.
Plumsteadville, Pennsylvania 18949

Contract No. 68-02-0652
Program Element No. 1HA327
ROAP No. 26ADZ

EPA Project Officer: John H. Margeson

Quality Assurance and Environmental Monitoring Laboratory
National Environmental Research Center
Research Triangle Park, North Carolina 27711

Prepared for

OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

June 1974

1a

This report has been reviewed by the Environmental Protection Agency and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the Agency, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.

Publication No. EPA-650/4-74-020

SRL 1317 13 0674

TABLE OF CONTENTS

		Page
1.0	INTRODUCTION	1-1
2.0	TEST PROGRAM	2-1
2.1	DESIGN OF TEST PROGRAM	2-1
2.2	DESCRIPTION OF TEST VARIABLES	2-4
2.3	ANALYTICAL PROCEDURES	2-5
2.4	ANALYTICAL SCHEDULE	2-6
3.0	RESULTS	3-1
3.1	ANALYSES OF TEST CYLINDERS	3-1
4.0	DATA ANALYSIS	4-1
4.1	DETERMINATION OF DETERIORATION RATE	4-1
4.2	REGRESSION ANALYSIS OF STABILITY DATA	4-3
4.3	SUMMARY OF EFFECTS OF TEST VARIABLES ON GAS STABILITY	4-19
4.4	EFFECT OF EXTREME STORAGE TEMPERATURES ON GAS STABILITY	4-20
4.5	EFFECT OF CYLINDER PRESSURE ON GAS STABILITY	4-24
4.6	EVALUATION OF THE EFFECT OF REGULATOR TYPE	4-25
5.0	DISCUSSION OF RESULTS	5-1
5.1	STABILITY OF CARBON MONOXIDE MIXTURES	5-1
5.2	STABILITY OF PROPANE MIXTURES	5-2
5.3	STORAGE OF CARBON MONOXIDE AND PROPANE MIXTURES	5-3
5.4	GENERAL COMMENTS	5-3
6.0	RECOMMENDATIONS	6-1
6.1	CARBON MONOXIDE IN NITROGEN MIXTURES	6-1
6.2	PROPANE IN AIR MIXTURES	6-2
7.0	ACKNOWLEDGEMENTS	7-1
8.0	REFERENCES	8-1

SRL 1317 13 0674

LIST OF ILLUSTRATIONS

		Page
FIGURE 4-1	CONCENTRATION - TIME TREND FOR CARBON MONOXIDE @ 50 PPM NOMINAL CONCENTRATION	4-2

LIST OF TABLES

TABLE 2-1	DESIGN OF VARIABLES FOR EACH OF TEN COMPONENT/ CONCENTRATION CYLINDER SETS	2-2
TABLE 2-2	DESIGNATION OF CONTROL CYLINDERS	2-3
TABLE 2-3	PROCEDURE FOR CARBON MONOXIDE ANALYSIS	2-7
TABLE 2-4	PROCEDURE FOR PROPANE ANALYSIS	2-7
TABLE 3-1	ANALYTICAL DATA FOR CARBON MONOXIDE AT 10 PPM	3-2
TABLE 3-2	ANALYTICAL DATA FOR CARBON MONOXIDE AT 50 PPM	3-3
TABLE 3-3	ANALYTICAL DATA FOR CARBON MONOXIDE AT 100 PPM	3-4
TABLE 3-4	ANALYTICAL DATA FOR CARBON MONOXIDE AT 500 PPM	3-5
TABLE 3-5	ANALYTICAL DATA FOR CARBON MONOXIDE AT 1000 PPM	3-6
TABLE 3-6	ANALYTICAL DATA FOR PROPANE AT 3 PPM	3-7
TABLE 3-7	ANALYTICAL DATA FOR PROPANE AT 10 PPM	3-8
TABLE 3-8	ANALYTICAL DATA FOR PROPANE AT 50 PPM	3-9
TABLE 3-9	ANALYTICAL DATA FOR PROPANE AT 100 PPM	3-10
TABLE 3-10	ANALYTICAL DATA FOR PROPANE AT 500 PPM	3-11
TABLE 4-1	PERCENT CHANGE IN CARBON MONOXIDE CONCENTRATION OVER A SIX-MONTH PERIOD	4-4
TABLE 4-2	PERCENT CHANGE IN PROPANE CONCENTRATION OVER A SIX-MONTH PERIOD	4-5
TABLE 4-3	VALUES ASSIGNED TO VARIABLES IN MULTIPLE REGRESSION MODEL	4-6
TABLE 4-4	EXAMPLE OF REGRESSION MATRIX FOR CARBON MONOXIDE AT 50 PPM NOMINAL CONCENTRATION	4-6
TABLE 4-5	ANALYSIS ON STABILITY OF CARBON MONOXIDE AT 10 PPM	4-9
TABLE 4-6	ANALYSIS ON STABILITY OF CARBON MONOXIDE AT 50 PPM	4-10
TABLE 4-7	ANALYSIS ON STABILITY OF CARBON MONOXIDE AT 100 PPM	4-11

LIST OF TABLES (CONTINUED)

		Page
TABLE 4-8	ANALYSIS ON STABILITY OF CARBON MONOXIDE AT 500 PPM	4-12
TABLE 4-9	ANALYSIS ON STABILITY OF CARBON MONOXIDE AT 1000 PPM	4-13
TABLE 4-10	ANALYSIS ON STABILITY OF PROPANE AT 3 PPM	4-14
TABLE 4-11	ANALYSIS ON STABILITY OF PROPANE AT 10 PPM	4-15
TABLE 4-12	ANALYSIS ON STABILITY OF PROPANE AT 50 PPM	4-16
TABLE 4-13	ANALYSIS ON STABILITY OF PROPANE AT 100 PPM	4-17
TABLE 4-14	ANALYSIS ON STABILITY OF PROPANE AT 500 PPM	4-18
TABLE 4-15	t-TEST ON HOT-CYCLE EFFECT FOR PROPANE AT 100 PPM NOMINAL CONCENTRATION	4-21
TABLE 4-16	SUMMARY OF HOT AND COLD STORAGE EFFECTS ON PROPANE STABILITY	4-23
TABLE 4-17	SUMMARY OF HOT AND COLD STORAGE EFFECTS ON CARBON MONOXIDE STABILITY	4-23
TABLE 4-18	EFFECT OF REDUCED CYLINDER PRESSURE ON STABILITY OF TRACE GASES	4-26
TABLE 4-19	COMPARISON OF REGULATOR TYPES IN ANALYSIS OF CARBON MONOXIDE	4-27
TABLE 4-20	COMPARISON OF REGULATOR TYPES IN ANALYSIS OF PROPANE	4-29

SET 1317 13 0674

ABSTRACT

The stability of gas mixtures of carbon monoxide in nitrogen and propane in air in high-pressure cylinders was investigated in 240 test cylinders over a six month period. The effect of several variables related to the preparation of the mixtures by the suppliers, the storage of the cylinders and their use by laboratories engaged in emissions measurements was studied. The variables included:

1. Cylinder wall material
2. Cylinder valve type
3. Cylinder preconditioning procedure
4. Concentration of carbon monoxide and propane
5. Purity of diluent nitrogen and air
6. Mixing procedure after blending
7. Temperature at which cylinders are stored
8. Cylinder pressure
9. Type of pressure-reducing regulator used

The concentration data obtained by periodic analysis of the 240 cylinders over the six month period were subjected to statistical analysis by multiple stepwise regression. The effects of the individual variables are discussed, and recommended practices for assuring stable mixtures of carbon monoxide in nitrogen and propane in air are presented.

1.0 INTRODUCTION

This report describes the work performed by Scott Research Laboratories, Inc. under EPA Contract No. 68-02-0652, "Development of Technical Specifications for Standard Gas-Diluent Mixtures for Use in Measurement of Mobile Source Emissions." The primary objective of this program was to develop technical specifications for producing stable gas mixtures of propane in air and carbon monoxide in nitrogen in high pressure gas cylinders. A stable mixture was defined as one in which the concentration of trace gas changed by no more than 3.0% from its original concentration over a six month test period.

In carrying out this project, Scott investigated the effect of variables related to the preparation of the gas mixtures by the suppliers as well as those related to the storage and use by laboratories engaged in emissions measurements. The first category included hardware type, preparation procedures and grade of diluent gas. In the latter category were hot and cold storage and cylinder regulator type.

This study was accomplished by utilizing 240 new cylinders purchased from the leading manufacturer of steel cylinders. The experimental design involved five concentration levels each of propane and carbon monoxide, two cylinder types, two valve types, three procedures for cylinder preparation, two mixing methods and three grades of diluent gas. Two thirds of the cylinders were subjected to hot and cold storage cycles with pressure reduction between each cycle. The remaining one-third served as controls. Each cylinder was analyzed by gas chromatography on a regular schedule.

The concentration data for each of the ten component/concentration level combinations were subjected to statistical analyses to estimate the effect of each variable on the concentration stability. This information was used to draw up recommendations for manufacturing and use specifications to assure stable mixtures.

2.0 TEST PROGRAM

2.1 DESIGN OF TEST PROGRAM

The test program was designed to study the effect of various material, cylinder preparation procedures and use procedures on the stability of five concentration levels each of carbon monoxide in nitrogen and propane in air. It was decided that 240 test cylinders, 120 each for carbon monoxide and propane, should be adequate to evaluate the effect of the variables and keep the magnitude of effort within practical limits. An examination of procedures followed by the various manufacturers of calibration gas mixtures resulted in the selection of five parameters at two or three levels each. These parameters and levels were believed to represent those materials and procedures in general use which could affect the stability of the mixtures.

For each of the ten component/concentration combinations, 24 cylinders were prepared according to the design given in Table 2-1. A detailed description of the variables is presented in the next section. The various levels of each variable were distributed among the 24 cylinders so as to produce an approximately balanced design which would facilitate subsequent data analysis.

Other variables were also studied. The effect of hot and cold storage was determined by exposing two-thirds or 16 of each set of 24 cylinders to elevated and lowered temperatures while maintaining the remaining one-third at constant ambient temperature. The latter group were termed control cylinders and they are identified in Table 2-2. The remaining 160 cylinders were each subjected to two 2-week storage periods at 95°F and two similar periods at 0°F. The storage sequence was either hot-cold-hot-cold or cold-hot-cold-hot. Approximately three weeks were allowed between successive storage periods.

It was also desired to determine the effect of reducing cylinder pressure on stability. For this purpose each of the cylinders subjected to hot and cold storage was reduced by 500 psi after each storage. Thus, the storage pressures were 2000 psi, 1500 psi, 1000 psi and 500 psi during the four successive storage periods. The control cylinders were maintained at approximately 2000 psi throughout the program.

SCOTT RESEARCH LABORATORIES, INC.

TABLE 2-1 DESIGN OF VARIABLES FOR EACH OF TEN COMPONENT/CONCENTRATION CYLINDER SETS

Cyl. No.	Cyl. Type	Valve Type	Pre-Conditioning	Mixing	Diluent Purity
1	2	1	1	1	1
2	2	1	2	1	1
3	2	1	3	1	1
4	2	1	1	2	1
5	2	1	2	2	1
6	2	1	3	2	1
7	2	2	1	2	2
8	2	2	2	1	2
9	2	2	3	2	2
10	2	2	1	1	3
11	2	2	3	1	3
12	2	2	2	2	3
13	1	1	1	1	2
14	1	1	2	1	3
15	1	1	3	1	2
16	1	1	1	2	3
17	1	1	2	2	2
18	1	1	3	2	3
19	1	2	1	1	1
20	1	2	2	1	1
21	1	2	3	1	1
22	1	2	1	2	1
23	1	2	2	2	1
24	1	2	3	2	1

KEY TO VARIABLES

Cylinder Type
1. Chrome-moly steel per DOT 3AA2015
2. Manganese Steel per DOT 3A2015

Valve Type
1. Packed brass with Teflon packing
2. Diaphragm packless brass

Preconditioning
1. Evaculation only
2. Evaculation + nitrogen flush + evacuation
3. Same as 2 with heat applied

Mixing
1. Mechanical
2. Thermal

Diluent Purity

	Air (C_3H_8)	Nitrogen (CO)
1.	Blended HC free	99.997% pure
2.	Blended N_2-O_2	99.7% pure; low moisture
3.	Water pumped	99.7% pure; high moisture

SRL 1317 13 0674

TABLE 2-2 DESIGNATION OF CONTROL CYLINDERS

Cyl No.*	Conc	Cyl No.*	Conc	Cyl No.*	Conc	Cyl No.*	Conc
1	A,B	7	B,D	13	B,E	19	D,E
2	C,D	8	C,E	14	C	20	C,D
3	E	9	D	15	D	21	A
4	A,D	10	A,E	16	A,C	22	B,C
5	C,E	11	B,C	17	B	23	A,E
6	A	12	B	18	A,B	24	D,E

*See Table 2-1

KEY TO CONCENTRATION LEVELS

Conc.	CO, ppm	Propane, ppm
A	10	3
B	50	10
C	100	50
D	500	100
E	1000	500

A final study involved the effect of cylinder regulator type. This part of the program is discussed in Section 4.6.

2.2 DESCRIPTION OF TEST VARIABLES

2.2.1 Cylinder Type

The two types of cylinders accounting for the large majority of carbon monoxide and propane mixtures in use are chrome-moly steel and manganese steel. The chrome-moly cylinders are manufactured to DOT Specification 3AA2015 as detailed in the Code of Federal Regulations (CFR) Title 49, Paragraph 178.37. The manganese steel cylinders are manufactured to DOT Specification 3A2015 as detailed in Paragraph 178.36 of the above CFR. All 240 test cylinders were purchased from the Taylor-Wharton Division of Harrisburg Steel Company, the leading supplier of such type cylinders. Prior to delivery to Scott, the cylinders were hydrostatically tested and steam cleaned by the manufacturer, as is the general practice.

2.2.2 Cylinder Valve Type

The two types of cylinder valves used were packed brass with Teflon packing and diaphragm packless brass. The packed brass valves were Superior Valve Co. Model 1200-E2. The packless brass were Superior Model 1250-E2.

2.2.3 Cylinder Preconditioning

The three types of preconditioning used were:

1. Evacuation only - evacuate cylinder as received to <0.1" Hg absolute.
2. Evacuation + nitrogen flush + evacuation - evacuate cylinder to <0.1" Hg, fill with pure nitrogen to 200 psi, evacuate to <0.1" Hg.
3. Same as 2 with heat applied - evacuate cylinder heated to 150°F for 15 minutes to <0.1" Hg, fill with pure nitrogen to 200 psi, reheat to 150°F for 15 minutes while evacuating to <0.1" Hg.

2.2.4 Mixing of Contents After Blending

Mixing is accomplished commercially by mechanical or thermal procedures or a combination of the two. It is of greater importance where high concentrations of low volatility gases are included in a mixture than in the current test program where the concentrations were relatively low, and carbon monoxide and propane both have relatively high vapor pressures.

Mechanical mixing was accomplished by placing the cylinders on rollers for 15 minutes. Thermal mixing was performed by heating the cylinder bottoms on a heated platen for ½ hour. The cylinder contents reached approximately 125°F.

2.2.5 Diluent Purity

Three levels of diluent purity were selected for both air and nitrogen. They are representative of the grades of diluent in current commercial use.

The nitrogen diluents were:

1. 99.997+ % (from liquid storage) dewpoint = -105°F.
2. 99.7% low moisture - dewpoint = -75°F.
3. 99.7% high moisture - dewpoint = -67°F.

Oxygen is the major impurity present in nitrogen of all grades. The air diluents were:

1. Blended hydrocarbon-free synthetic air (O_2 = 20.4%, N_2 = 79.6%) THC = 0.1 ppm, dewpoint = -105°F.
2. Blended synthetic air (O_2 = 20.4%, N_2 = 79.6%), THC = 5 ppm-C, dewpoint = -85°F.
3. Water pumped air, THC = 3 ppm-C, dewpoint = -75°F.

2.3 ANALYTICAL PROCEDURES

All test cylinders were analyzed by gas chromatography. Carbon monoxide was determined using a Varian Aerograph Model 1520 with a helium ionization detector at the conditions shown in Table 2-3. Concentrations were calculated by comparing the peak heights of the standard and test cylinders.

Propane was determined using a Perkin-Elmer Model 900 with a flame ionization detector. The instrument conditions are given in Table 2-4. Concentrations were calculated from peak areas measured by an Infotronics Model CRS-100 Integrator. Total hydrocarbon analyses were obtained with a Beckman Model 108A Flame Ionization Detector.

Except for the different instruments, the schemes for analysis of propane and carbon monoxide were identical. Each test cylinder at a particular level was analyzed using a single standard cylinder of CO in N_2 or propane in air as the reference. Each analysis consisted of three to five injections of gas from each of the test and standard cylinders. The two cylinders were sampled on an alternate basis to eliminate any possibility of errors due to time trends in instrument response.

The cylinder standards were rechecked every two to four weeks by comparison to primary standards in glass flasks. The primary standards were prepared by a procedure used by Scott in analyzing close tolerance gas mixtures sold commercially. This procedure has been shown to have a high degree of accuracy and repeatability. The specific details are proprietary. The procedure basically involves the injection of a known volume of trace gas of known purity from a calibrated gas-tight syringe into a calibrated 5-liter glass flask. The glass flask is then pressurized to a measured pressure of approximately 0.5 atmosphere gauge.

The overall analysis scheme produced an accuracy of ±1% for propane and ±2% for carbon monoxide. The error for carbon monoxide was higher because the carbon monoxide peak fell on the tail of the nitrogen peak. This necessitated construction of a baseline which increased error especially at low concentration levels.

2.4 ANALYTICAL SCHEDULE

Each cylinder was analyzed within several days after preparation and at various intervals thereafter over the six month test period. The control cylinders were analyzed five to seven times and the others 11 to 15 times. This included analyses before and after each hot or cold storage period. The analyses after hot and cold storage were performed within several hours of removal from storage.

SRL 1317 13 0674

TABLE 2-3 PROCEDURE FOR CARBON MONOXIDE ANALYSIS

Instrument: Varian Aerograph Model 1520
Detector: Helium Ionization
Column: 10' Molecular Sieve
Temperature: 75°C
Carrier Gas: Helium @ 50 psi
Sample Size: 0.25 to 1 cc

TABLE 2-4 PROCEDURE FOR PROPANE ANALYSIS

Instrument: Perkin-Elmer Model 900
Detector: Flame Ionization
Column: 9' x 1/8" Porapak Q
Temperature: 150°C
Carrier Gas: Helium @ 50 psi
Flame Gas: Hydrogen @ 20 psi
Combustion Gas: Oxygen @ 50 psi
Sample Size: 2 cc

SRL 1317 13 0674

3.0 RESULTS

3.1 ANALYSES OF TEST CYLINDERS

The analytical data for the 120 carbon monoxide test cylinders are shown in Tables 3-1 through 3-5. The corresponding data for the propane test cylinders are shown in Tables 3-6 through 3-10. The cylinder age is the number of days from the date of blending to the date of analysis. The concentrations are in parts per million. The data shown in these tables provided the base for the data analysis described in Section 4.0.

TABLE 3-1 ANALYTICAL DATA FOR CARBON MONOXIDE AT 10 PPM

Cyl. A11349
Code 21111*

Age	Conc.
13	10.39
31	10.81
45	10.33
91	10.55
113	10.72
136	10.41
153	10.39
168	10.35
183	10.56
205	10.40
223	10.41

Cyl. A11359
Code 21211

Age	Conc.
17	10.08
31	9.66
78	10.56
99	10.18
122	10.00
139	9.86
154	9.98
169	10.13
191	10.10
209	10.18

Cyl. A11369
Code 21311

Age	Conc.
20	10.16
54	10.27
75	10.69
97	10.81
120	10.16
137	10.43
152	10.59
167	10.69
189	10.53
208	10.40

Cyl. A11379
Code 21121

Age	Conc.
26	9.97
54	10.40
75	10.80
97	10.79
120	10.15
137	10.81
152	11.01
167	10.64
176	10.43
189	10.66
208	10.51

Cyl. A11389
Code 21221

Age	Conc.
22	10.30
50	10.29
71	10.82
93	10.59
116	10.25
133	10.39
148	10.41
163	10.71
172	10.20
204	10.41

Cyl. A11399
Code 21321

Age	Conc.
22	9.91
50	10.06
72	10.57
143	10.16
157	10.10
184	10.03

Cyl. A11409
Code 22122

Age	Conc.
7	9.09
20	9.38
36	9.27
58	9.33
142	9.75
169	9.48
203	9.89

Cyl. A11419
Code 22212

Age	Conc.
0	10.37
17	10.45
52	10.79
68	10.66
91	10.22
108	10.44
123	10.44
138	10.32
160	10.27
179	10.39

Cyl. A11429
Code 22322

Age	Conc.
2	10.88
16	10.63
47	10.72
64	10.97
86	10.53
107	10.49
118	10.59
133	10.44
155	10.40
174	10.16
201	10.43

Cyl. A11439
Code 22113

Age	Conc.
2	10.83
15	10.79
41	11.22
121	11.04
148	11.02
199	11.09

Cyl. A11449
Code 22313

Age	Conc.
5	9.25
28	10.26
39	9.65
54	10.00
76	9.36
94	10.03
97	9.39
109	9.39
124	9.27
150	9.65
164	9.73
182	9.76

Cyl. A11459
Code 22223

Age	Conc.
6	13.52
28	13.57
39	13.60
54	13.98
76	13.29
94	13.29
109	12.88
124	13.41
150	12.87
164	13.32
182	13.32

Cyl. A11469
Code 11112

Age	Conc.
12	9.15
25	9.36
41	9.45
63	9.67
147	9.46
174	9.14
208	9.72

Cyl. A11479
Code 11213

Age	Conc.
6	10.71
39	11.53
53	11.58
69	11.42
91	10.76
109	11.02
112	10.99
124	10.84
139	10.60
165	10.93
179	11.04
197	11.01

Cyl. A11489
Code 11312

Age	Conc.
2	10.51
35	10.51
49	10.73
65	10.88
87	10.69
105	10.20
108	10.42
120	9.99
135	9.11
141	9.21
161	9.62
175	9.76
193	9.65

Cyl. A11499
Code 11123

Age	Conc.
1	11.52
37	12.44
48	12.24
63	12.11
85	11.54
103	11.82
106	11.63
118	11.75
133	11.14
139	11.65
159	11.52
173	11.64
191	11.54

Cyl. A11509
Code 11222

Age	Conc.
5	10.09
28	10.47
39	10.43
54	10.41
76	9.62
94	9.65
109	9.55
124	8.81
130	8.93
150	9.06
164	9.23
182	9.37

Cyl. A11519
Code 11323

Age	Conc.
6	11.27
28	11.57
69	10.94
125	10.88
133	11.23
165	11.32

Cyl A11529
Code 12111

Age	Conc.
2	10.54
16	10.55
47	10.57
64	10.38
86	10.30
103	10.26
118	10.18
133	10.08
155	9.66
174	9.99
201	10.04

Cyl. A11539
Code 12211

Age	Conc.
2	10.46
15	9.91
41	10.43
121	10.06
148	10.15
182	10.28

Cyl. A11549
Code 12311

Age	Conc.
2	9.74
51	8.02
64	8.01
135	7.08
149	7.58
175	7.65

Cyl. A11559
Code 12121

Age	Conc.
3	10.09
38	9.40
53	9.11
68	7.97
90	7.05
108	7.26
123	6.57
138	6.41
144	4.59
164	5.15
178	5.30
196	5.00

Cyl. A11569
Code 12221

Age	Conc.
6	10.42
41	10.72
90	10.15
104	9.95
131	9.91
165	10.09

Cyl. A11579
Code 12321

Age	Conc.
5	11.20
28	11.82
39	11.38
54	11.95
76	11.34
94	11.30
109	11.37
124	11.22
150	10.93
164	11.21
182	11.30

* See page 2-2 for explanation of code.

SCOTT RESEARCH LABORATORIES, INC.

TABLE 3-2 ANALYTICAL DATA FOR CARBON MONOXIDE AT 50 PPM

Cyl. A11350
Code 21111*

Age	Conc.
8	50.86
30	51.61
43	50.20
87	51.69
106	51.91
129	52.32
146	51.24
161	51.57
176	51.83
199	51.12
218	51.94

Cyl. A11360
Code 21211

Age	Conc.
3	49.96
16	50.96
31	49.83
74	51.38
132	50.80
188	51.09
212	51.55

Cyl. A11370
Code 21311

Age	Conc.
8	50.30
21	53.94
54	52.20
72	53.01
130	51.71
186	53.90
222	53.79

Cyl. A11380
Code 21121

Age	Conc.
8	51.05
21	52.51
54	51.65
72	52.37
90	52.05
113	52.37
130	51.50
145	51.97
160	51.64
183	51.88
202	52.42

Cyl. A11390
Code 21221

Age	Conc.
7	50.64
23	49.94
51	51.71
68	52.78
86	52.41
109	52.58
126	51.46
141	51.35
156	52.24
163	52.28
179	51.92
198	52.43

Cyl. A11400
Code 21321

Age	Conc.
7	51.05
23	50.52
51	52.24
68	52.54
86	52.26
109	52.18
126	52.19
141	51.61
156	52.81
163	51.67
179	51.27
198	52.31

Cyl. A11410
Code 22122

Age	Conc.
2	60.32
20	59.54
37	59.17
56	59.97
83	59.52
98	58.61
113	58.37
136	58.54
154	57.87
168	59.24
184	58.68
213	58.68

Cyl A11420
Code 22212

Age	Conc.
3	52.49
17	51.77
53	52.65
73	52.76
88	51.88
103	51.53
126	51.88
144	51.13
158	52.14
174	52.51
203	52.92

Cyl. A11430
Code 22322

Age	Conc.
1	53.54
16	52.83
48	53.83
68	54.05
83	52.80
98	52.52
121	52.80
139	52.21
153	53.05
169	53.06
201	53.13

Cyl. A11440
Code 22113

Age	Conc.
2	48.42
16	48.08
42	49.05
64	49.43
87	48.32
104	47.89
119	48.88
135	47.84
142	48.55
157	47.11
174	49.91
197	49.94

Cyl. A11450
Code 22313

Age	Conc.
5	49.37
41	49.89
90	49.31
124	48.51
133	50.71
165	50.19

Cyl. A11460
Code 22223

Age	Conc.
7	53.10
31	53.76
42	54.00
59	54.03
74	52.55
89	52.85
112	53.30
129	54.18
145	53.78
159	55.17
185	55.13
189	55.56

Cyl. A11470
Code 11112

Age	Conc.
7	55.40
25	55.04
42	54.97
61	55.44
147	54.08
172	54.19
208	55.49

Cyl. A11480
Code 11213

Age	Conc.
6	52.40
41	52.44
47	52.97
62	52.65
85	51.71
102	51.44
117	52.46
133	51.37
140	51.60
158	51.13
174	53.50
195	53.01
203	52.65

Cyl. A11490
Code 11312

Age	Conc.
2	51.94
37	52.65
43	52.00
58	51.39
81	50.80
98	50.55
113	51.22
129	49.17
136	49.36
154	48.70
170	50.63
191	50.08
199	49.89

Cyl. A11500
Code 11123

Age	Conc.
1	47.01
22	48.38
78	47.59
113	47.72
138	47.49
174	49.46
210	49.51

Cyl. A11510
Code 11222

Age	Conc.
5	50.58
41	50.44
90	49.09
124	47.95
133	49.24
165	49.31

Cyl. A11520
Code 11323

Age	Conc.
7	53.96
31	54.35
42	54.71
59	53.80
74	53.56
89	53.09
112	53.78
129	54.03
145	53.62
159	53.69
185	53.55
189	53.63

Cyl. A11530
Code 12111

Age	Conc.
1	52.30
16	51.13
48	53.07
68	52.68
83	52.27
98	51.72
121	52.25
139	51.38
146	52.06
153	52.67
169	52.77
201	53.72

Cyl. A11540
Code 12211

Age	Conc.
2	52.04
16	51.46
42	51.48
64	50.33
87	49.03
104	49.50
119	49.73
135	48.22
142	48.88
157	48.18
174	50.84
197	49.78

Cyl. A11550
Code 12311

Age	Con.
2	50.94
23	51.54
52	50.98
69	50.93
84	50.17
99	49.79
122	50.10
139	50.72
155	50.57
195	50.72
199	50.67

Cyl. A11560
Code 12121

Age	Conc.
3	52.65
39	53.00
56	52.43
73	52.36
88	52.10
103	50.96
108	52.18
126	51.79
143	51.43
159	51.70
173	51.75
199	52.07

Cyl. A11570
Code 12221

Age	Conc.
6	51.58
41	52.05
90	51.20
124	50.59
133	52.10
165	52.35
175	52.00

Cyl. A11580
Code 12321

Age	Conc.
6	54.26
41	53.63
90	53.82
124	52.51
133	54.42
157	54.66

* See page 2-2 for explanation of code.

TABLE 3-3 ANALYTICAL DATA FOR CARBON MONOXIDE AT 100 PPM

Cyl. A11351
Code 21111*

Age	Conc.
7	109.15
29	108.23
50	112.21
91	110.27
106	109.50
129	110.17
146	110.16
161	109.36
176	109.56
199	111.19
218	108.69

Cyl. A11361
Code 21211

Age	Conc.
6	107.20
17	106.65
36	109.36
77	108.53
92	107.40
115	107.00
132	107.70
147	106.71
162	107.12
185	107.44
204	106.75

Cyl. A11371
Code 21311

Age	Conc.
6	111.70
54	111.95
19	111.35
75	109.90
180	111.62
204	109.72

Cyl. A11381
Code 21121

Age	Conc.
6	108.17
20	109.80
54	111.95
75	110.20
90	111.10
113	110.03
130	109.22
145	108.39
160	108.91
183	109.68
202	108.28

Cyl. A11391
Code 21221

Age	Conc.
4	111.60
30	113.30
71	111.27
98	109.53
113	112.37
128	109.76
151	109.84
169	110.80
183	112.71
199	109.37

Cyl. A11401
Code 21321

Age	Conc.
4	108.20
29	111.13
71	108.07
86	109.93
109	107.83
126	108.08
141	106.67
156	106.63
179	107.22
198	106.01
221	107.60

Cyl. A11411
Code 22122

Age	Con.
2	106.02
24	106.50
58	107.70
83	106.73
161	107.44
185	106.06
213	107.07

Cyl. A11421
Code 22212

Age	Conc.
4	105.17
18	106.85
83	106.53
151	107.25
187	106.48
229	106.43

Cyl. A11431
Code 22322

Age	Conc.
2	114.64
16	112.40
55	110.27
70	110.47
93	110.15
110	110.23
125	110.09
141	111.79
166	113.46
180	110.84
203	112.25

Cyl. A11441
Code 22113

Age	Conc.
3	103.73
17	105.93
43	105.09
64	102.84
87	103.75
104	103.21
119	102.88
135	104.32
160	102.30
176	104.70
197	105.20

Cyl. A11451
Code 22313

Age	Conc.
0	91.41
26	92.88
42	91.64
60	92.69
74	91.35
89	91.11
112	91.46
130	94.31
138	95.13
146	91.99
159	94.27
185	94.21
192	93.77

Cyl. A11461
Code 22223

Age	Conc.
11	108.60
42	109.27
84	108.05
123	111.57
157	109.86
201	111.21

Cyl. A11471
Code 11112

Age	Conc.
7	105.50
29	106.50
63	109.15
88	106.75
103	108.67
118	107.72
141	106.43
159	107.19
173	108.78
189	105.07
228	106.45

Cyl. A11481
Code 11213

Age	Conc.
6	110.37
41	109.00
70	108.45
139	109.97
175	109.96

Cyl. A11491
Code 11312

Age	Conc.
2	111.20
37	108.33
43	107.67
58	107.25
81	106.43
98	106.68
113	106.56
129	106.27
154	108.09
168	104.95
191	105.20
203	105.41

Cyl. A11501
Code 11123

Age	Conc.
1	107.17
35	108.95
51	107.47
69	106.77
83	105.76
98	106.41
121	106.71
138	108.60
155	105.60
168	106.60
194	107.62
201	107.46

Cyl. A11511
Code 11222

Age	Conc.
0	107.33
26	106.60
42	103.55
60	105.10
74	104.10
89	100.34
94	98.91
112	99.40
130	103.04
138	104.01
146	99.97
159	99.12
185	98.48
192	98.05

Cyl. A11521
Code 11323

Age	Conc.
11	109.33
27	108.47
46	107.10
61	105.53
84	103.72
101	104.89
118	106.37
138	103.61
157	101.49
171	101.96
185	100.61
192	100.97

Cyl. A11531
Code 12111

Age	Conc.
0	111.07
16	108.90
78	108.10
146	109.37
182	108.84
224	108.68

Cyl. A11541
Code 12211

Age	Conc.
3	104.90
17	104.57
43	105.70
64	101.67
87	103.45
104	101.17
107	101.76
119	101.59
135	101.22
160	102.15
197	102.81
209	101.99

Cyl. A11551
Code 12311

Age	Conc.
2	103.57
36	103.41
52	101.40
70	100.87
84	98.99
99	99.46
122	99.43
139	100.97
148	100.06
156	99.58
169	99.85
195	99.71
202	99.07

Cyl. A11561
Code 12121

Age	Conc.
4	109.73
56	109.53
98	106.68
137	110.81
171	107.51

Cyl. A11571
Code 12221

Age	Conc.
6	116.83
27	114.70
46	112.67
61	111.65
84	108.47
101	109.65
118	109.98
131	109.22
157	108.61
171	110.52
185	109.86
192	110.75

Cyl. A11581
Code 12321

Age	Conc.
6	111.20
27	111.03
55	109.65
124	109.01
160	108.71
201	109.29

* See page 2-2 for explanation of code.

SCOTT RESEARCH LABORATORIES, INC.

TABLE 3-4 ANALYTICAL DATA FOR CARBON MONOXIDE AT 500 PPM

Cyl. A11352
Code 21111 *

Age	Conc.
9	552.80
29	554.45
50	542.86
93	543.00
111	550.20
126	553.30
141	556.62
164	550.90
181	545.72
196	549.73
211	556.66

Cyl. A11362
Code 21211

Age	Conc.
6	537.77
21	541.35
56	526.80
79	527.35
170	529.00
192	531.86
233	530.55

Cyl. A11372
Code 213112

Age	Conc.
5	555.57
19	552.25
54	551.00
77	555.87
95	551.20
110	558.67
125	554.06
148	550.92
165	546.53
180	550.81
195	553.58

Cyl. A11382
Code 21121

Age	Conc.
5	522.47
19	518.15
54	518.95
77	522.73
168	525.10
190	529.12

Cyl. A11392
Code 21221

Age	Conc.
3	503.80
29	510.45
63	511.50
164	521.20
184	519.82
225	520.72

Cyl. A11402
Code 21321

Age	Conc.
3	544.97
18	541.60
63	543.53
88	535.01
105	546.73
120	541.07
135	535.95
158	537.25
175	532.29
190	540.86
206	538.30
234	542.88

Cyl. A11413
Code 22122

Age	Conc.
1	540.50
24	530.50
59	530.70
76	536.00
91	541.45
106	534.74
129	532.67
146	530.41
161	532.17
176	529.51
211	535.01

Cyl. A11422
Code 22212

Age	Conc.
4	532.57
18	542.40
55	541.33
80	545.75
96	545.40
110	544.07
133	544.01
150	536.76
165	545.83
181	542.95
209	550.70

Cyl. A11432
Code 22322

Age	Conc.
2	523.40
16	516.13
106	515.66
148	509.76
259	516.13

Cyl. A11442
Code 22113

Age	Conc.
3	537.57
27	549.40
86	546.76
142	542.09
191	549.13

Cyl. A11452
Code 22313

Age	Conc.
0	536.67
32	531.35
46	542.40
62	538.20
84	537.39
101	537.18
117	538.06
131	543.09
154	550.00
171	545.88
185	545.39
193	545.98

Cyl. A11462
Code 22223

Age	Conc.
11	514.32
48	519.60
83	518.18
125	511.44
174	522.84

Cyl. A11472
Code 11112

Age	Conc.
6	531.60
29	535.70
64	524.90
81	528.17
96	534.20
111	526.68
134	530.35
151	522.73
154	531.17
166	519.91
181	535.20
216	529.40

Cyl. A11482
Code 11213

Age	Conc.
6	550.03
27	555.93
61	549.73
76	556.80
99	542.29
116	549.41
120	549.93
132	543.55
146	552.52
169	548.18
186	559.92
201	560.78
208	556.56

Cyl. A11492
Code 11312

Age	Conc.
2	513.62
23	514.60
80	515.06
136	503.13
185	513.34

Cyl. A11502
Code 11123

Age	Conc.
1	522.37
20	517.50
58	521.27
75	522.46
91	521.67
105	520.29
128	519.16
145	530.27
152	517.19
160	527.65
175	535.38
195	534.68

Cyl. 11512
Code 11222

Age	Conc.
0	538.67
32	533.60
46	538.40
62	532.30
84	535.97
101	536.39
117	530.51
131	544.14
137	544.66
154	550.88
171	545.04
185	547.26
193	546.52

Cyl. A11522
Code 11323

Age	Conc.
11	546.77
35	540.63
46	544.53
61	546.40
84	541.50
101	538.43
117	534.87
131	545.37
137	553.50
154	525.15
171	554.72
186	554.37
193	549.68

Cyl. A11532
Code 12111

Age	Conc.
0	537.43
16	543.73
71	542.77
134	548.53
154	548.75
197	545.26
215	545.14

Cyl. A11542
Code 12211

Age	Conc.
3	527.43
27	530.20
52	528.83
69	539.43
84	530.64
99	533.79
122	534.90
139	525.83
154	535.32
170	535.41
198	541.02

Cyl. A11552
Code 12311

Age	Conc.
2	501.60
21	499.40
59	503.40
76	499.61
92	499.00
106	496.68
129	494.33
146	504.67
153	495.65
161	509.55
176	518.81
196	507.88

Cyl. A11562
Code 12121

Age	Conc.
4	545.23
41	541.57
49	537.10
66	542.23
82	543.88
96	531.17
103	527.56
119	530.12
136	522.17
151	529.06
167	513.51
195	520.64

Cyl. A11572
Code 12221

Age	Conc.
4	613.20
33	588.63
46	595.07
62	601.17
84	590.47
101	587.10
117	589.24
131	592.50
154	599.77
171	599.64
186	608.62
195	613.13

Cyl. A11582
Code 12321

Age	Conc.
4	534.43
33	532.50
46	539.30
62	536.33
84	534.55
101	529.98
117	524.13
131	532.76
154	532.30
171	534.15
186	543.35
195	557.89

* See page 2-2 for explanation of code.

TABLE 3-5 ANALYTICAL DATA FOR CARBON MONOXIDE AT 1000 PPM

Cyl. A11353
Code 21111 *

Age	Conc.
10	1133.00
29	1121.00
43	1122.92
94	1128.67
184	1135.97
204	1117.83
240	1126.32

Cyl. A11363
Code 21211

Age	Conc.
6	1105.33
22	1079.00
55	1093.00
80	1091.33
105	1089.67
120	1102.33
134	1095.02
157	1083.02
176	1096.95
189	1093.08
206	1092.20

Cyl. A11373
Code 21311

Age	Conc.
5	1064.50
20	1037.00
53	1058.50
78	1059.33
95	1052.67
110	1057.00
125	1065.00
148	1055.78
165	1049.78
180	1057.08
195	1045.83

Cyl. A11383
Code 21121

Age	Conc.
5	1080.00
20	1066.50
53	1089.50
78	1082.50
95	1081.00
110	1085.00
125	1091.57
148	1082.67
165	1076.96
180	1095.95
195	1070.11

Cyl. A11393
Code 21221

Age	Conc.
3	1089.00
18	1089.00
63	1095.67
164	1088.27
184	1074.94

Cyl. A11403
Code 21321

Age	Conc.
3	1126.67
18	1140.00
63	1115.00
136	1114.76
179	1113.62
220	1104.90

Cyl. A11412
Code 22122

Age	Conc.
1	1123.33
27	1115.00
59	1112.00
77	1109.52
91	1116.50
106	1124.67
129	1118.33
146	1113.59
161	1128.34
176	1093.66
212	1107.77

Cyl. A11423
Code 22212

Age	Conc.
4	1125.00
18	1121.67
111	1131.03
154	1123.19
201	1124.84

Cyl. A11433
Code 22322

Age	Conc.
5	1055.67
19	1075.00
85	1080.50
149	1084.69
184	1092.46
224	1056.54

Cyl. A11443
Code 22113

Age	Conc.
6	1116.00
28	1112.33
52	1112.33
69	1108.50
84	1117.33
99	1110.94
122	1097.91
139	1133.86
142	1120.37
154	1103.63
171	1079.76
198	1114.59

Cyl. A11453
Code 22313

Age	Conc.
3	1097.37
34	1113.33
49	1099.00
66	1112.10
81	1103.12
96	1103.99
119	1113.50
136	1094.95
153	1108.14
166	1134.31
187	1111.15
194	1125.39

Cyl. A11463
Code 22223

Age	Conc.
10	1111.33
34	1114.67
49	1099.33
66	1111.38
81	1105.45
96	1104.15
119	1124.35
136	1103.51
151	1149.72
166	1120.25
187	1113.72
194	1130.31

Cyl. A11473
Code 11112

Age	Conc.
6	1093.33
32	1095.50
64	1090.33
82	1091.33
96	1086.00
111	1098.15
134	1087.57
153	1084.00
166	1103.47
181	1065.13
217	1071.10

Cyl. A11483
Code 11213

Age	Conc.
5	1095.33
27	1087.67
77	1099.00
141	1092.08
176	1117.71
211	1092.52

Cyl. A11493
Code 11312

Age	Conc.
1	1100.00
23	1082.33
38	1087.00
57	1081.00
72	1094.00
86	1089.47
109	1077.37
128	1090.32
141	1088.64
158	1091.45
192	1082.08
205	1082.59

Cyl. A11503
Code 11123

Age	Conc.
2	1024.50
20	1013.00
71	1033.50
135	1014.37
175	1027.53
205	1055.76

Cyl. A11513
Code 11222

Age	Conc.
3	1122.00
34	1129.00
49	1116.00
66	1139.34
81	1117.78
96	1119.08
119	1131.19
136	1110.95
153	1127.16
166	1151.86
187	1128.11
194	1121.82

Cyl. A11523
Code 11323

Age	Conc.
10	1115.33
34	1107.67
49	1092.33
66	1117.03
81	1097.72
96	1104.69
119	1118.98
136	1088.28
151	1140.68
166	119.73
187	1103.74
196	1120.46

Cyl. A11533
Code 12111

Age	Conc.
1	1083.67
19	1098.00
57	1106.33
75	1104.67
90	1110.84
105	1093.38
128	1077.47
145	1120.73
148	1105.51
160	1081.70
174	1082.20
204	1090.56

Cyl. A11543
Code 12211

Age	Conc.
6	1117.33
28	1118.67
52	1115.00
69	1116.00
84	1123.47
99	1113.01
122	1093.18
139	1134.94
142	1120.73
154	1106.42
17	1086.70
198	1115.94

Cyl. A11553
Code 12311

Age	Conc.
3	1138.00
21	1123.00
72	1135.50
136	1115.28
176	1137.48
206	1152.85

Cyl. A11563
Code 12121

Age	Conc.
4	1124.00
41	1118.00
48	1124.67
66	1122.00
81	1133.24
96	1107.73
103	1123.74
119	1087.52
136	1133.14
139	1124.94
151	1102.62
165	1117.45
195	1119.42

Cyl. A11573
Code 12221

Age	Conc.
4	989.37
34	993.33
49	988.73
67	992.63
81	999.28
96	996.84
119	1001.92
136	989.07
151	972.70
166	1028.25
187	996.60
194	1006.35

Cyl. A11583
Code 12321

Age	Conc.
4	1140.67
34	1146.67
49	1138.67
67	1145.36
81	1150.29
96	1144.05
119	1167.85
136	1140.11
151	1129.65
166	1169.04
187	1153.58

* See page 2-2 for explanation of code.

TABLE 3-6 ANALYTICAL DATA FOR PROPANE AT 3 PPM

Cyl. A11344
Code 21111*

Age	Conc.
27	3.34
43	3.32
56	3.27
79	3.27
106	3.22
111	3.27
120	3.24
127	3.24
146	3.20
160	3.26
176	3.29
197	3.29

Cyl. A11354
Code 21211

Age	Conc.
13	3.17
29	3.14
42	3.12
65	3.09
92	3.10
106	3.08
113	3.07
132	3.02
136	3.08
146	3.12
148	3.14
162	3.11
183	3.24
188	3.16
203	3.12

Cyl. A11364
Code 21311

Age	Conc.
4	3.26
26	3.20
39	3.19
54	3.23
90	3.14
95	3.16
104	3.16
111	3.10
130	3.10
144	3.19
146	3.20
160	3.20
181	3.20
201	3.16

Cyl. A11374
Code 21121

Age	Conc.
4	3.17
26	3.19
39	3.12
54	3.13
90	3.13
104	3.15
111	3.17
130	3.10
134	3.16
144	3.17
160	3.20
166	3.21
181	3.19
201	3.11

Cyl. A11384
Code 21221

Age	Conc.
23	3.18
37	3.17
51	3.20
73	3.20
100	3.17
123	3.19
140	3.22
154	3.20
170	3.19
190	3.23
191	3.20

Cyl. A11394
Code 21321

Age	Conc.
3	3.24
23	3.25
37	3.23
59	3.20
80	3.21
101	3.23
123	3.25
144	3.09
148	3.26
164	3.24
185	3.24
204	3.24

Cyl. A1140
Code 22122

Age	Conc.
7	3.32
13	3.25
27	3.19
49	3.19
66	3.20
114	3.24
136	3.27
167	3.27
175	3.25
189	3.25
196	3.27

Cyl. A11414
Code 22212

Age	Conc.
3	3.23
17	3.23
33	3.20
53	3.23
75	3.16
98	3.22
115	3.19
129	3.24
145	3.24
165	3.25
185	3.18
200	3.23

Cyl. A11424
Code 22322

Age	Conc.
5	3.22
20	3.22
34	3.19
51	3.21
68	3.20
83	3.23
98	3.22
121	3.24
139	3.22
152	3.20
168	3.18
202	3.16

Cyl. A11434
Code 22113

Age	Conc.
1	3.14
15	3.14
29	3.13
45	3.10
70	3.16
93	3.14
115	3.17
154	3.15
175	3.20
177	3.14
196	3.19

Cyl. A11444
Code 22313

Age	Conc.
3	3.28
24	3.27
41	3.24
59	3.29
68	3.28
89	3.27
111	3.28
129	3.29
143	3.23
144	3.29
160	3.20
164	3.30
186	3.28
188	3.28

Cyl. A11454
Code 22223

Age	Conc.
4	3.30
24	3.27
41	3.25
59	3.26
68	3.28
89	3.27
111	3.31
132	3.27
143	3.25
160	3.27
188	3.30

Cyl. A11464
Code 11112

Age	Conc.
5	3.47
18	3.20
32	3.14
48	3.20
68	3.21
88	3.19
110	3.21
131	3.28
134	3.04
138	3.22
152	3.22
175	3.18
186	3.15
189	3.21
194	3.21

Cyl. A11474
Code 11213

Age	Conc.
5	3.13
20	3.14
41	3.14
62	3.14
85	3.16
102	3.17
116	3.17
132	3.11
152	3.17
153	3.15
172	3.07
174	3.09
193	3.12

Cyl. A11484
Code 11312

Age	Conc.
1	3.13
16	3.12
37	3.12
58	3.09
81	3.07
98	3.11
112	3.10
128	3.13
148	3.17
149	3.10
168	3.06
170	3.05
183	3.11
189	3.09

Cyl. A11494
Code 11123

Age	Conc.
2	3.14
20	3.08
35	3.12
54	3.10
69	3.08
84	3.13
104	3.16
107	3.15
125	3.07
133	3.14
138	3.10
154	3.11

Cyl. A11504
Code 11222

Age	Conc.
4	3.15
24	3.14
41	3.12
59	3.15
68	3.19
89	3.19
111	3.19
129	3.19
143	3.11
144	3.17
160	3.13
164	3.17
178	3.16
179	3.12
186	3.16

Cyl. A11514
Code 11323

Age	Conc.
19	3.32
20	3.32
28	3.29
48	3.31
95	3.34
111	3.35
132	3.34
151	3.34
188	3.34

Cyl. A11524
Code 12111

Age	Conc.
5	3.16
20	3.15
34	3.18
51	3.13
68	3.18
84	3.19
98	3.12
100	3.19
121	3.14
139	3.19
152	3.16
168	3.20
169	3.14
189	3.14

Cyl. A11534
Code 12211

Age	Conc.
0	3.03
15	2.98
29	2.97
45	2.96
70	3.02
93	3.02
115	3.10
120	3.03
154	3.02
175	3.04
195	3.00
196	3.05

Cyl. A11544
Code 12311

Age	Conc.
3	3.02
21	2.98
36	3.02
58	2.99
101	2.78
105	3.03
121	3.00
142	3.03
161	3.03
196	3.10
197	3.05

Cyl. A11554
Code 12121

Age	Conc.
0	3.11
19	3.08
39	3.07
68	2.85
84	3.01
89	3.06
91	3.05
109	3.15
112	3.08
123	3.13
138	3.13
159	3.04
160	3.10
178	3.10
185	3.09

Cyl. A11564
Code 12221

Age	Conc.
4	3.13
24	3.10
41	3.12
63	3.08
82	3.14
95	3.17
124	3.18
146	3.15
151	3.16
167	3.14
188	3.16

Cyl. A11574
Code 12321

Age	Conc.
4	3.16
24	3.14
41	3.11
59	3.14
68	3.16
89	3.16
111	3.17
129	3.15
143	3.09
144	3.12
160	3.14
188	3.12

* See page 2-2 for explanation of code.

TABLE 3-7 ANALYTICAL DATA FOR PROPANE AT 10 PPM

Cyl. A11345
Code 21111*

Age	Conc.
27	10.90
43	10.85
56	10.80
79	10.80
106	10.72
120	10.75
127	10.75
146	10.95
150	10.79
160	10.79
176	10.95
182	10.72
184	10.64
197	10.87

Cyl. A11355
Code 21211

Age	Conc.
13	10.90
29	10.80
42	10.80
65	10.80
84	10.84
104	10.73
126	10.75
147	10.86
150	11.33
154	10.90
168	10.80
191	10.90

Cyl. A11365
Code 21311

Age	Conc.
4	10.97
26	11.00
39	10.95
54	11.00
77	10.97
96	10.95
117	10.75
118	10.99
138	10.98
160	11.05
181	11.00
200	11.09

Cyl. A11375
Code 21121

Age	Conc.
4	11.10
26	11.13
39	11.15
54	11.17
90	11.07
95	11.04
104	11.03
111	11.04
130	10.91
144	11.10
160	11.14
181	11.08
201	11.11

Cyl. A11385
Code 21221

Age	Conc.
3	10.87
23	10.90
37	10.90
51	10.90
73	10.88
100	10.83
123	10.88
140	10.94
154	10.93
170	10.96
190	10.98
210	11.11

Cyl. A11395
Code 21321

Age	Conc.
2	11.00
23	11.00
37	11.10
59	11.13
86	11.03
100	11.03
107	11.06
126	10.96
130	11.17
140	11.13
156	11.21
164	11.01
177	11.12
197	11.11

Cyl. A11405
Code 22122

Age	Conc.
7	10.47
13	10.30
27	10.40
49	10.40
71	10.38
85	10.39
92	10.38
111	10.35
125	10.44
141	10.36
162	10.46
182	10.42

Cyl. A11415
Code 22212

Age	Conc.
3	11.07
17	10.97
33	10.95
53	10.93
75	10.95
98	10.95
115	10.98
129	10.99
145	11.15
165	11.05
185	11.29

Cyl. A11425
Code 22322

Age	Conc.
5	11.03
20	11.00
34	11.00
51	11.02
68	10.97
83	10.89
98	11.06
121	11.06
139	11.02
152	10.99
168	11.11
169	11.02
202	11.14

Cyl. A11435
Code 22113

Age	Conc.
1	10.53
15	10.60
29	10.55
45	10.53
62	10.51
77	10.42
92	10.59
115	10.57
133	10.57
146	10.65
162	10.66
196	10.62

Cyl. A11445
Code 22313

Age	Conc.
3	10.80
24	10.74
41	10.71
63	10.58
66	10.79
82	10.78
124	10.74
146	10.80
167	10.72
186	10.77
188	10.84

Cyl. A11455
Code 22223

Age	Conc.
4	10.90
24	10.89
41	10.86
59	10.84
68	10.87
89	10.83
111	10.72
129	10.91
143	10.90
160	10.92
188	10.94

Cyl. A11465
Code 11112

Age	Conc.
5	10.63
18	10.60
32	10.60
48	10.60
68	10.59
88	10.56
110	10.63
131	10.64
152	10.61
180	10.65
194	10.66

Cyl. A11475
Code 11213

Age	Conc.
5	10.97
20	10.97
41	11.02
62	11.01
85	10.96
102	11.06
116	11.02
132	11.09
152	11.14
172	11.05
193	11.00

Cyl. A11485
Code 11312

Age	Conc.
1	10.87
16	10.80
37	10.85
58	10.79
81	10.73
98	10.86
100	10.89
112	10.89
128	10.94
148	10.90
168	10.88
183	10.89

Cyl. A11495
Code 11123

Age	Conc.
2	10.80
20	10.80
35	10.76
57	10.87
78	10.77
100	10.96
104	10.86
120	10.73
141	10.81
160	10.82

Cyl. A11505
Code 11222

Age	Conc.
3	17.40
24	17.37
41	17.21
82	17.49
124	17.46
146	17.35
167	17.37
186	17.37

Cyl. A11515
Code 11323

Age	Conc.
20	15.48
28	15.51
48	15.48
66	15.51
73	15.23
77	15.62
98	15.63
117	15.64
136	15.58
147	15.73
151	15.66
166	15.67
188	15.58

Cyl. A11525
Code 12111

Age	Conc.
5	10.90
20	10.90
34	10.90
51	10.91
68	10.83
83	10.77
98	10.92
100	10.92
121	10.94
139	10.94
152	10.90
168	10.95
189	10.84
190	11.02

Cyl. A11535
Code 12211

Age	Conc.
0	10.82
15	10.80
29	10.80
45	10.84
62	10.72
77	10.70
92	10.85
115	10.86
133	10.79
149	10.90
162	10.91
195	10.86

Cyl. A11545
Code 12311

Age	Conc.
3	10.65
21	10.78
36	10.77
55	10.66
70	10.58
85	10.73
108	10.75
126	10.79
139	10.75
155	10.85
156	10.83
196	10.82

Cyl. A11555
Code 12121

Age	Conc.
0	11.07
19	11.10
39	11.12
73	11.02
82	11.09
103	11.15
125	10.98
143	11.14
157	11.21
174	11.23
185	11.06

Cyl. A11565
Code 12221

Age	Conc.
3	10.70
24	10.77
41	10.62
62	10.53
82	10.66
124	10.75
146	10.86
151	10.79
167	10.83
188	10.73

Cyl. A11575
Code 12321

Age	Conc.
4	11.20
24	11.26
41	11.15
63	11.15
82	11.18
124	11.38
129	11.37
146	11.14
151	11.25
167	11.15
188	11.08

See page 2-2 for explanation of code.

SCOTT RESEARCH LABORATORIES, INC.

TABLE 3-8 ANALYTICAL DATA FOR PROPANE AT 50 PPM

Cyl. A11346
Code 21111 *

Age	Conc.
15	52.20
27	51.77
43	52.35
56	52.10
79	52.30
111	51.77
112	51.93
126	52.26
141	52.53
162	52.76
182	52.14
211	52.59

Cyl. A11356
Code 21211

Age	Conc.
13	53.10
29	53.00
42	53.05
65	52.77
97	52.75
98	52.80
112	53.06
127	53.30
148	53.74
168	53.30
183	53.06
197	53.33

Cyl. A11366
Code 21311

Age	Conc.
4	53.27
20	52.85
39	52.55
54	52.40
76	52.80
95	52.48
138	52.61
160	53.34
181	52.85
200	53.15

Cyl. A11376
Code 21121

Age	Conc.
4	54.60
20	54.75
39	54.65
54	54.45
76	54.70
97	54.45
118	55.03
137	54.88
152	55.58
155	55.86
167	54.99
189	55.07

Cyl. A11386
Code 21221

Age	Conc.
3	53.70
23	53.25
37	52.90
51	52.70
72	53.30
92	52.95
107	53.31
128	53.46
148	53.90
162	53.68
177	53.25
198	53.49

Cyl. A11396
Code 21321

Age	Conc.
2	52.73
23	52.40
37	52.65
59	52.00
91	52.25
92	52.03
106	52.50
121	52.60
122	52.87
142	53.03
144	53.00
162	52.23
164	52.76
191	52.59

Cyl. A11406
Code 22122

Age	Conc.
1	51.43
13	51.20
27	51.10
49	51.05
66	51.25
91	51.35
114	51.15
136	52.75
139	51.79
170	51.55
175	51.69
196	51.81

Cyl. A11416
Code 22212

Age	Conc.
3	53.30
17	53.45
33	53.30
52	53.51
73	53.37
95	54.11
116	53.93
137	53.87
160	53.80
179	54.10
188	53.95

Cyl. A11426
Code 22322

Age	Conc.
5	53.70
20	53.30
34	53.40
51	53.60
77	53.42
99	53.41
118	54.39
121	54.32
132	53.58
147	53.74
168	54.08
187	53.93

Cyl. A11436
Code 22113

Age	Conc.
1	60.05
15	59.45
29	59.70
45	59.85
71	59.89
92	60.18
112	60.95
115	60.62
126	60.43
141	59.96
162	60.34
181	60.44
202	59.75

Cyl. A11446
Code 22313

Age	Conc.
3	52.40
21	52.70
41	52.76
61	52.65
62	52.96
80	52.32
103	53.07
116	52.69
132	53.15
150	53.21
171	52.91
187	52.88

Cyl. A11456
Code 22223

Age	Conc.
4	53.27
21	53.40
41	53.40
63	53.94
82	53.41
124	53.61
146	53.69
167	53.58
187	53.95

Cyl. A11466
Code 11112

Age	Conc.
5	52.92
18	52.75
32	52.70
48	52.47
67	53.07
83	52.72
104	53.11
123	53.23
138	53.61
141	53.24
153	52.87
175	53.11
194	53.17

Cyl. A11476
Code 11213

Age	Conc.
5	55.95
20	55.85
41	56.16
63	56.01
84	55.87
88	56.20
106	56.63
126	56.05
147	56.45
166	56.18
200	56.07

Cyl. A11486
Code 11312

Age	Conc.
1	52.92
16	52.63
37	53.12
65	53.07
86	52.85
106	53.79
109	53.68
120	53.16
135	53.54
156	53.67
175	53.18
177	53.32
183	53.44

Cyl. A11496
Code 11123

Age	Conc.
2	52.92
20	53.05
35	53.10
63	52.94
84	53.68
86	52.96
104	53.87
118	53.44
133	53.35
154	53.26
173	53.41

Cyl. A11506
Code 11222

Age	Conc.
3	50.90
21	51.20
41	51.40
61	51.42
77	51.64
80	51.12
103	51.30
116	51.15
132	51.50
150	51.62
171	50.82
172	51.45
185	51.78

Cyl. A11516
Code 11323

Age	Conc.
19	53.30
28	53.25
48	53.53
66	53.49
73	53.47
98	53.97
117	52.94
136	53.44
147	53.90
166	53.11
167	53.60
187	53.17
188	53.27

Cyl. A11526
Code 12111

Age	Conc.
5	52.80
20	52.50
34	52.95
51	52.85
76	52.83
99	53.08
121	54.73
124	53.21
160	53.18
181	53.26
189	53.21

Cyl. A11536
Code 12211

Age	Conc.
0	52.72
15	52.50
29	52.65
45	52.83
71	52.86
92	53.22
112	53.47
126	53.19
141	53.08
162	53.74
163	53.33
181	53.27
202	53.23

Cyl. A11546
Code 12311

Age	Conc.
3	51.77
21	51.95
36	52.40
64	52.18
85	52.63
105	52.85
119	52.20
134	52.44
155	52.27
174	52.30
195	52.27

Cyl. A11556
Code 12121

Age	Conc.
0	54.07
19	53.95
38	54.03
59	54.28
81	54.74
102	54.65
123	54.33
146	54.74
164	53.72
165	54.58
173	49.41
185	54.46

Cyl. A11566
Code 12221

Age	Conc.
3	52.80
24	53.00
41	52.98
61	53.55
80	53.53
101	53.31
103	53.15
116	53.16
132	53.52
171	53.43
187	53.45

Cyl. A11576
Code 12321

Age	Conc.
4	54.15
21	54.35
41	54.14
63	54.73
82	54.36
124	54.08
146	54.16
167	54.25
187	54.66

* See page 2-2 for explanation of code.

TABLE 3-9 ANALYTICAL DATA FOR PROPANE AT 100 PPM

Cyl. A11347
Code 21111*

Age	Conc.
15	105.00
27	109.00
43	108.00
56	108.50
79	108.00
111	107.75
112	107.70
126	108.51
141	107.00
142	107.68
211	107.17

Cyl. A11357
Code 21211

Age	Conc.
2	107.33
13	107.67
29	108.00
42	107.00
65	107.00
83	106.27
104	106.40
126	106.18
147	106.09
168	105.40
19	105.47
210	106.95

Cyl. A11367
Code 21311

Age	Conc.
4	108.00
20	108.50
39	108.00
54	107.00
76	106.65
97	106.56
118	106.31
137	106.12
152	106.63
167	107.04
189	106.11
208	106.07

Cyl. A11377
Code 21121

Age	Conc.
4	108.33
20	109.00
39	109.00
54	109.00
76	108.50
96	108.44
117	107.66
138	107.97
160	107.14
181	108.31
186	107.64
200	108.04

Cyl. A11387
Code 21221

Age	Conc.
3	107.33
23	107.50
37	107.50
51	107.00
72	106.70
92	106.61
113	105.79
134	105.37
156	105.82
182	105.34
196	105.93
219	107.16

Cyl. A11397
Code 21321

Age	Conc.
2	102.33
23	102.00
37	102.00
59	101.50
91	101.25
92	101.20
106	100.47
121	100.23
142	101.51
144	100.95
162	101.90
191	100.64

Cyl. A11407
Code 22122

Age	Conc.
1	103.33
13	103.00
27	102.50
49	102.50
76	102.55
77	102.50
91	102.15
106	101.76
127	102.36
147	101.87
176	101.19
184	102.20

Cyl. A11417
Code 22212

Age	Conc.
3	103.00
17	102.00
33	102.00
52	102.33
81	102.00
91	101.81
110	101.55
131	101.70
150	100.87
164	102.22
165	101.34
181	101.95
188	102.27

Cyl. A11427
Code 22322

Age	Conc.
5	108.00
20	108.00
34	108.00
51	106.90
76	107.04
99	105.44
100	105.69
103	104.69
121	107.06
124	107.11
160	106.67
181	106.87
208	106.66

Cyl. A11437
Code 22113

Age	Conc.
1	102.00
15	102.00
29	101.57
45	100.73
70	101.10
93	100.97
115	101.34
154	100.86
175	101.19
202	100.76

Cyl. A11447
Code 22313

Age	Conc.
3	105.65
21	105.20
41	105.30
61	104.92
80	105.00
103	104.71
116	105.51
132	105.20
150	104.73
171	104.91
187	105.13

Cyl. A11457
Code 22223

Age	Conc.
4	108.00
21	107.55
41	107.75
82	106.83
124	106.84
146	106.95
167	107.50
187	107.05

Cyl. A11467
Code 11112

Age	Conc.
5	107.00
18	106.50
32	106.00
48	106.00
68	105.70
83	105.90
104	104.85
105	105.12
123	105.22
138	105.68
153	105.82
175	105.78
194	105.93

Cyl. A11477
Code 11213

Age	Conc.
5	105.00
20	105.00
41	105.05
81	103.87
88	103.63
113	104.92
116	103.83
132	104.74
151	104.73
162	104.81
181	104.89
200	104.49

Cyl. A11487
Code 11312

Age	Conc.
1	107.00
16	107.33
37	106.55
59	106.95
81	106.58
102	106.77
122	106.49
143	106.67
162	106.16
183	106.27

Cyl. A11497
Code 11123

Age	Conc.
2	105.50
20	105.00
35	105.27
62	104.46
72	104.68
91	104.20
112	104.37
131	104.56
145	104.82
162	104.92

Cyl. A11507
Code 11222

Age	Conc.
3	102.00
21	101.20
41	101.50
61	100.00
62	100.68
80	100.53
101	101.50
103	100.07
116	101.68
132	101.31
150	101.03
171	100.46
172	101.00
185	100.89

Cyl. A11517
Code 11323

Age	Conc.
19	104.67
28	104.63
48	102.60
66	103.42
73	103.79
98	105.05
103	104.20
117	104.31
136	104.51
147	105.73
166	105.58
187	105.01

Cyl. A11527
Code 12111

Age	Conc.
5	107.33
20	106.50
34	106.75
51	106.65
76	106.35
99	106.30
121	106.12
160	106.47
181	106.11
189	106.43

Cyl. A11537
Code 12211

Age	Conc.
0	105.00
15	105.00
29	105.00
45	103.75
70	104.89
80	103.92
99	103.69
120	103.47
139	104.36
153	104.70
154	103.07
170	104.07
202	104.26

Cyl. A11547
Code 12311

Age	Conc.
3	102.50
21	103.00
36	102.35
63	102.22
73	102.69
92	102.07
113	102.43
132	102.51
146	102.75
163	102.31
195	102.79

Cyl. A11557
Code 12121

Age	Conc.
0	108.00
19	108.00
38	107.60
67	107.60
96	107.39
117	107.08
150	108.01
151	105.96
152	106.79
167	106.75
185	107.37

Cyl. A11567
Code 12221

Age	Conc.
3	102.00
21	101.95
41	102.40
59	102.00
68	101.99
89	101.88
111	102.41
129	101.81
143	101.95
160	102.30
187	101.73

Cyl. A11577
Code 12321

Age	Conc.
4	109.60
21	108.85
41	108.97
66	108.29
73	108.80
98	109.48
117	108.40
136	108.78
147	108.89
166	110.06
167	109.20
187	108.40

* See page 2-2 for explanation of code.

SCOTT RESEARCH LABORATORIES, INC.

TABLE 3-10 ANALYTICAL DATA FOR PROPANE AT 500 PPM

Cyl. A11348
Code 21111

Age	Conc.
15	556.33
27	558.00
43	556.50
56	555.50
79	556.33
97	554.40
118	555.20
140	556.97
161	558.81
182	559.40
205	558.75

Cyl. A11358
Code 21211

Age	Conc.
2	529.67
13	525.67
29	529.50
42	529.00
65	528.67
97	526.70
98	528.07
112	525.10
127	529.79
148	533.54
150	530.84
168	529.80
197	527.89

Cyl. A11368
Code 21311

Age	Conc.
4	523.00
20	521.50
39	523.00
54	519.00
76	522.50
97	521.34
118	522.00
137	523.14
152	524.91
167	521.96
189	520.78
208	523.77

Cyl. A11378
Code 21121

Age	Conc.
4	530.67
26	532.00
39	532.67
54	536.00
76	532.00
97	532.89
118	529.61
137	533.74
152	536.75
155	534.90
167	532.64
189	534.53
208	537.81

Cyl. A11388
Code 21221

Age	Conc.
3	547.67
23	552.00
37	555.00
51	553.00
72	553.00
92	552.70
113	552.67
134	549.79
162	556.20
177	552.64
196	548.32
219	560.19

Cyl. A11398
Code 21321

Age	Conc.
3	600.00
23	605.00
37	603.50
59	602.00
79	604.63
101	602.01
122	607.32
144	607.20
164	610.70
185	607.32
204	605.40

Cyl. A11408
Code 22122

Age	Conc.
1	538.00
13	538.00
27	537.00
49	534.00
76	536.03
77	538.10
91	535.42
106	540.44
127	540.56
147	540.77
162	538.31
176	538.17

Cyl. A11418
Code 22212

Age	Conc.
3	517.33
17	517.50
33	515.00
52	518.47
73	518.06
95	516.12
116	519.89
137	522.10
160	517.83
179	516.94
188	519.73

Cyl. A11428
Code 22322

Age	Conc.
5	557.33
20	555.00
34	555.00
51	556.85
99	553.84
121	556.93
160	556.18
181	555.11
202	551.43

Cyl. A11438
Code 22113

Age	Conc.
1	528.50
15	525.00
29	525.00
45	527.70
70	524.06
80	525.54
99	526.15
120	529.30
139	526.48
153	525.39
170	526.14
196	521.10
203	526.84

Cyl. A11448
Code 22313

Age	Conc.
3	564.00
21	564.55
41	567.07
61	564.87
80	563.65
103	567.66
116	565.54
132	563.54
150	574.29
151	567.13
171	567.45
186	566.93
187	566.12

Cyl. A11458
Code 22223

Age	Conc.
4	561.00
21	562.55
41	562.00
66	562.34
73	561.84
117	562.91
136	563.56
147	566.00
166	564.59
187	564.17

Cyl. A11468
Code 11112

Age	Conc.
5	538.33
18	538.50
32	541.50
48	536.33
67	538.27
83	537.39
104	539.47
123	540.21
138	541.54
153	539.53
175	544.30
194	541.34

Cyl. A11478
Code 11213

Age	Conc.
5	548.25
20	546.00
41	547.63
63	544.67
84	549.45
106	549.20
126	550.89
147	549.70
166	545.25
194	545.94

Cyl. A11488
Code 11312

Age	Conc.
1	526.33
16	521.00
37	524.95
72	519.33
73	528.30
91	518.84
112	525.75
127	522.90
143	523.82
161	524.70
182	524.61
183	524.92

Cyl. A11498
Code 11123

Age	Conc.
2	513.25
20	512.50
35	514.45
57	512.29
78	515.48
100	517.82
141	517.55
160	513.56

Cyl. A11508
Code 11222

Age	Conc.
3	510.67
21	512.55
41	513.03
61	511.46
80	512.29
98	515.44
116	513.92
132	512.43
150	519.38
151	512.53
171	514.10
186	513.56

Cyl. A11518
Code 11323

Age	Conc.
19	547.67
28	548.80
48	543.48
66	546.71
73	546.40
98	550.94
117	544.04
136	545.70
147	552.19
150	550.55
166	550.01
188	543.43
199	542.61

Cyl. A11528
Code 12111

Age	Conc.
5	527.33
20	525.50
34	523.00
63	526.73
84	527.02
103	526.46
118	529.75
133	525.58
155	523.76
174	527.32
189	533.56
190	527.55

Cyl. A11538
Code 12211

Age	Conc.
0	540.75
15	539.50
29	538.50
45	540.10
70	538.02
80	542.52
99	539.63
120	542.76
139	539.02
153	540.57
170	544.65
174	538.44
196	542.10

Cyl. A11548
Code 12311

Age	Conc.
3	517.25
21	515.00
36	517.70
58	516.91
63	516.91
79	516.95
101	520.52
121	521.05
142	521.76
161	516.43
196	519.11

Cyl. A11558
Code 12121

Age	Conc.
0	541.00
19	537.00
38	540.32
67	538.74
96	541.62
117	540.43
150	539.35
167	533.09
171	540.92
185	538.03

Cyl. A11568
Code 12221

Age	Conc.
3	525.00
21	526.20
41	528.73
59	526.24
68	529.96
89	529.54
111	534.35
132	531.53
143	521.25
144	527.07
160	534.16
172	526.47
179	526.52
188	532.56
189	523.51

Cyl. A11578
Code 12321

Age	Conc.
4	534.75
21	537.97
41	536.90
66	539.78
73	537.93
98	540.22
117	541.18
136	538.85
147	540.18
166	543.43
188	538.33

* See pages 2-2 for explanation of code.

4.0 DATA ANALYSIS

The primary objective of the program was to develop specifications for producing stable gas mixtures of propane in air and carbon monoxide in nitrogen in high pressure gas cylinders. A stable mixture was defined as one in which the concentration of trace gas changes by no more than 3% from its original concentration over a six month test period.

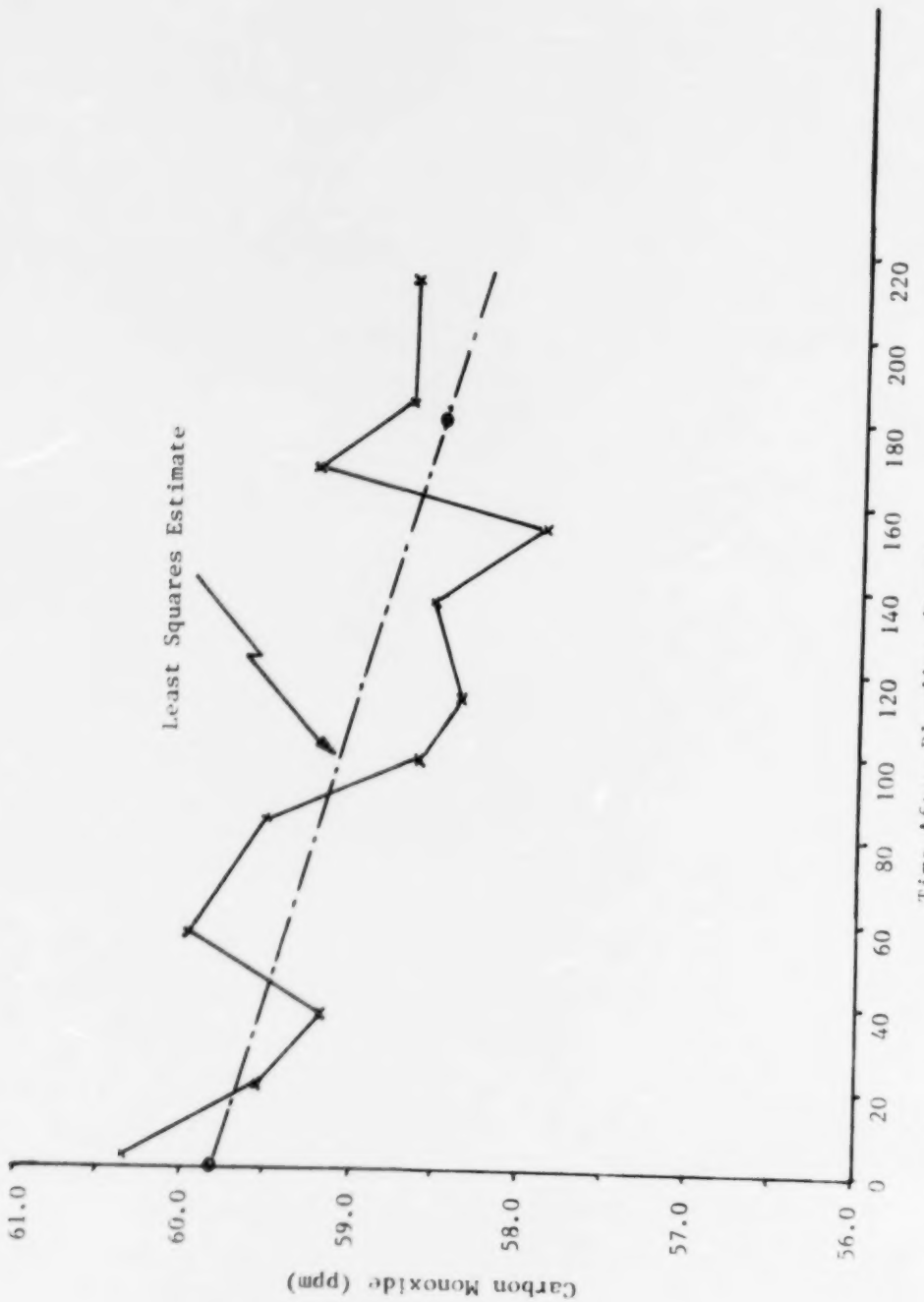
The test cylinder concentration data shown in Tables 3-1 through 3-10 were analyzed statistically to determine the influence of each of the five hardware type and mixture preparation variables on stability of the cylinder mixtures. Separate analyses were performed for each of the ten component/concentration range combinations. The test variables, which were described in detail in Section 2.2, included cylinder type, valve type, preconditioning procedure, mixing procedure after blending, and purity of diluent gas.

4.1 DETERMINATION OF DETERIORATION RATE

The first step in the data analysis was to determine the average rate of change of each of the 240 test cylinders during the six month test period. Plots of the concentration determinations as a function of age produced irregular lines as illustrated in Figure 4-1, which shows the concentration-time variations for carbon monoxide at a 50 ppm nominal concentration. The illustration is for a manganese steel cylinder using a diaphragm packless brass valve, preconditioned by evacuation only, mixed by thermal agitation and diluted with 99.7% pure nitrogen with a low moisture content. In order to establish some definite quantitative measure of stability, a general trend in the time-deterioration characteristic of the trace gas had to be developed. The method of least squares was selected for this purpose. The broken line in Figure 4-1 illustrates the time-concentration regressed least squares estimate of trace gas concentration deterioration. Once this least squares estimate was obtained, the percent change in concentration over the six month period was computed according to Equation 1.

SRL 1317 13 0674

FIGURE 4-1 CONCENTRATION - TIME TREND FOR CARBON MONOXIDE @ 50 PPM NOMINAL CONCENTRATION



SRL 1317 13 0674

$$\% \text{ change} = \frac{C_i - C_f}{C_i} \times 100 \qquad (1)$$

where: C_i = Concentration at time of preparation.

C_f = Final concentration at end of 180 days.

This percent change in concentration was used as a measure of the stability of the trace gases. The percent changes for each of the 24 cylinders at each of five levels of carbon monoxide and propane are shown in Table 4-1 and 4-2 respectively.

4.2 REGRESSION ANALYSIS OF STABILITY DATA

The effect of five qualitative variables on the stability of trace gases as defined by their percent change in concentration over a six month period was assessed. The technique of multiple regression was selected as the method for estimating the magnitude and effect that these variables had on the percent change in concentration of trace gases in high pressure cylinders. Since these are qualitative variables in the sense that they are not defined on a continuous scale of measurement but consist instead of groups of discrete items, the method outlined in Reference 1 was utilized.

In this method, where a variable consists of only two types of items (e.g., cylinder type, valve type or mixing) the value '-1' is assigned to one of the items and '+1' to the other. When appropriate, interactions between this variable and any other variable may also be incorporated in the selection of the regression model. Of course, in the final regression equation that is selected, this variable can only take the values ±1. When there are three types of items in a variable (e.g., preconditioning or diluent purity), a quadratic term of this variable must be included in the model, i.e., the equation must have an x_i and an x_i^2 term (x_i being the three-item variable). The three items of this variable are given the values: -1, 0, +1. In the interpretation of the final model the x_i and x_i^2 terms must be considered jointly.

Table 4-3 shows the values assigned to each of the items comprising the five variables studied. Using these values, a regression matrix of

SRL 1317 13 0674

TABLE 4-1 PERCENT CHANGE IN CARBON MONOXIDE CONCENTRATION OVER A SIX-MONTH PERIOD

Cyl. No.	Cylinder Code	10 ppm	50 ppm	100 ppm	500 ppm	1000 ppm
1	21111	-1.03	1.32	0.07	0.35	-0.04
2	21211	0.97	1.91	-0.68	-0.96	-0.04
3	21311	1.99	2.93	-0.89	-0.55	0.13
4	21121	3.43	0.48	-0.98	1.53	0.43
5	21221	-0.15	2.65	-1.45	2.62	-0.81
6	21321	-0.06	1.39	-2.43	-1.15	-1.86
7	22122	6.08	-2.19	0.29	-0.17	-0.81
8	22212	-1.87	0.11	-1.20	1.39	0.13
9	22322	-4.65	-1.05	-0.64	-0.81	0.62
10	22113	1.66	1.08	-0.40	0.85	-0.79
11	22313	-0.78	1.19	2.80	2.20	1.64
12	22223	-3.38	3.68	2.31	0.84	1.85
13	11112	1.59	-0.91	-0.11	-0.65	-1.58
14	11213	-2.83	0.12	0.30	0.90	0.79
15	11312	-12.79	-5.07	-3.48	-0.94	-0.51
16	11123	-4.75	3.48	-0.34	2.26	1.93
17	11222	-14.85	-4.20	-7.35	2.48	0.58
18	11323	-1.93	-1.11	-7.27	0.98	1.03
19	12111	-6.43	1.58	-0.88	1.02	-0.45
20	12211	-0.73	-4.80	-2.56	1.47	-1.00
21	12311	-22.47	-0.98	-3.25	1.78	0.93
22	12121	-52.24	-2.03	-1.26	-5.25	-0.78
23	12221	-6.79	0.69	-4.57	1.08	1.32
24	12321	-3.84	0.18	-1.87	1.79	1.10

SRL 1317 13 0674

TABLE 4-2 PERCENT CHANGE IN PROPANE CONCENTRATION OVER A SIX-MONTH PERIOD

Cyl. No.	Cylinder Code	3 ppm	10 ppm	50 ppm	100 ppm	500 ppm
1	21111	-1.68	-0.45	0.83	0.04	0.55
2	21211	0.52	1.30	0.76	-1.45	0.29
3	21311	-1.55	0.53	0.40	-1.53	0.19
4	21121	0.37	-0.45	1.53	-1.01	0.68
5	21221	1.02	1.24	0.71	-1.21	0.55
6	21321	-0.25	0.78	0.51	-1.23	0.88
7	22122	0.69	0.32	1.25	-1.32	0.52
8	22212	0.08	1.75	1.35	-0.66	0.44
9	22322	-0.95	0.80	1.17	-1.14	-0.41
10	22113	1.49	0.98	0.86	-0.80	-0.27
11	22313	-0.12	0.47	0.93	-0.39	0.60
12	22223	0.17	0.38	0.80	-0.69	0.63
13	11112	-2.67	0.43	1.01	-0.81	0.73
14	11213	-0.97	0.81	0.50	-0.16	0.03
15	11312	-0.84	0.82	1.06	-0.76	0.14
16	11123	0.18	0.27	1.02	-0.65	0.70
17	11222	-0.02	0.18	0.57	-0.45	0.66
18	11323	1.26	1.57	-0.13	1.63	-0.14
19	12111	0.23	0.51	1.40	-0.60	0.62
20	12211	1.47	0.84	1.47	-0.83	0.36
21	12311	2.13	1.38	0.63	0.03	0.65
22	12121	1.62	0.66	-1.87	-0.89	-0.34
23	12221	1.76	1.39	1.00	-0.12	0.21
24	12321	-0.80	-0.19	0.19	-0.03	0.86

TABLE 4-3 VALUES ASSIGNED TO VARIABLES IN MULTIPLE REGRESSION MODEL

	Variable		Type of Item	Description	Value Assigned
	Cylinder Material	x_2	1	Chrome moly steel	-1
			2	Manganese steel	+1
	Valve Type	x_3	1	Packed brass with Teflon	-1
			2	Diaphragm packless brass	+1
	Pre-Conditioning	x_4	1	Evacuation only	-1
			2	Evacuation + N_2 flush + evacuation	0
			3	same as 2 with heat applied	+1
	Mixing	x_5	1	Mechanical	-1
			2	Thermal	+1
Carbon Monoxide:	Diluent Purity	x_6	1	99.997% pure N_2	-1
			2	99.7% pure N_2; low moisture	0
			3	99.7% pure N_2; high moisture	+1
Propane:	Diluent Purity	x_6	1	Blended HC-free air	-1
			2	Blended N_2-O_2	0
			3	Water pumped	+1

TABLE 4-4 EXAMPLE OF REGRESSION MATRIX FOR CARBON MONOXIDE AT 50 PPM NOMINAL CONCENTRATION

Obs. No.	Dep. Variable	Independent Variables						
	% Change y	Cyl. Type x_2	Valve Type x_3	Precon-ditioning x_4	Mixing x_5	Dil. Purity x_6	x_4^2	x_6^2
1	1.32	1	-1	-1	-1	-1	1	1
2	1.91	1	-1	0	-1	-1	0	1
3	2.93	1	-1	1	-1	-1	1	1
4	0.48	1	-1	-1	1	-1	1	1
.	.	.	.	.	.	.	.	.
.	.	.	.	.	.	.	.	.
.	.	.	.	.	.	.	.	.
24	0.18	-1	1	1	1	-1	1	1

the type shown in Table 4-4 was developed for each of the five nominal concentration levels of carbon monoxide and propane.

Standard analysis of variance methods could have been used on the data collected. Such methods often require balanced statistically designed experiments and unfortunately, the precise requirements of these designs cannot always be achieved in practice. In such cases, regression analysis techniques are very suitable and provide the necessary answers on the magnitude and effect that independent variables have on the response of a dependent variable.

One of the most important steps in any regression analysis is the selection of the "best" independent variables for inclusion in the final regression equation. Once the appropriate regression model has been ascertained, analysis of variance techniques can be applied to determine what effect the independent variables have on the response of the dependent variable. The dependent variable (y), in our case, is the percent change in concentration over a six month period. The independent variables are described in Table 4-3. Since we are considering the effect of more than one independent variable, a multiple linear regression model was employed. The basic form of a multiple regression model is:

$$y = a_o + a_1(x_2) + a_2(x_3) + a_3(x_4) + \ldots$$

where y is the dependent variable, x_i are the independent variables, a_o is the intercept and the a_i terms are the regression coefficients we are interested in estimating. Although a regression model can incorporate all the independent variables (and functions of these independent variables), not all the independent variables necessarily explain the variation in the dependent variable. The selection of the "significant" independent variables, therefore, is accomplished by a statistical test known as the F-test. To do this, we determine whether the estimate of the variance (mean sum of squares) which is attributable to regression, is significantly greater than the estimate of the variance which deviates from regression. (The square root of the latter quantity is termed the standard error of estimate.) The ratio of the above two variances is termed the F-ratio and it is then compared against standard F-tables. If the calculated F is

significnatly greater than the tabulated F (at a given P% probability level, usually 1% to 10%), we can assert with a P% risk that the variate under consideration contributes significantly to the regression and that there probably exists a genuine relationship between the independent variable and the dependent variable. The contribution of the sum of squares due to the "non-significant" variables is incorporated in the residual term, since the association (if any) between the "non-significant" independent variables and the dependent variable can be attributed only to chance or random causes.

A number of techniques are available for selection of the "best" regression model. All of these techniques, however, use the F-test for assessing the significance of a variable. The model which is finally selected should be simple; incorporate those variables which contribute significantly in explaining the variation in the data; and, reflect the situation as observed from practical experience. For the purposes of this study the technique of Multiple Stepwise Regression was employed in the selection of the best set of independent variables. In this technique, variables are added into the model one by one but, at each stage, any variable which is already included in the model but whose extra sum of squares contribution has declined to a non-significant level is eliminated. However, this variable may possibly be brought in again at a latter stage. Selection stops when all unused variables are non-significant and all the included variables are significant.

With the percent change in concentration for each level of carbon monoxide and propane as a dependent variable, the regression variables shown in Table 4-4 were subject to the above technique. Besides the variables shown in Table 4-4, interaction and second-order variable effects were also studied, but no significant effects could be determined. The results of the multiple stepwise regression and the corresponding analysis or variance are presented in Tables 4-5 through 4-9 for carbon monoxide and in Tables 4-10 through 4-14 for propane.

SRL 1317 13 0674

TABLE 4-5 ANALYSIS ON STABILITY OF CARBON MONOXIDE AT 10 PPM

Multiple Stepwise Regression:

Only cylinder type (x_2) is significant.*

Regression Model:

$y = a_o + a_1 (x_2)$ $\quad$ y = % change

$a_o = -5.2445$

$a_1 = 5.4271$

Standard Error of Estimate = 10.63%

Analysis of Variance:

Source	Sum of Squares	Degrees of Freedom	Mean Squares	F-Value	% Contribution
x_2	706.88	1	706.88	6.26	22.14
Residual	2485.50	22	112.98		77.86
Total	3192.38	23			100.00

Standard $F_{1,22} = 4.30$ @ 0.05 probability point.

*Note: Diluent purity most likely has an effect on the stability of carbon monoxide in cylinders at this concentration; but the presence of extreme values (see data points 15, 17, 21 and 22 in Table 4-1) associated with cylinders of type 1 (chrome-moly steel) apparently tends to mask this effect.

SRL 1317 13 0674

TABLE 4-6 ANALYSIS ON STABILITY OF
CARBON MONOXIDE AT 50 PPM

Multiple Stepwise Regression:

Cylinder type (x_2) and diluent purity (x_6) are the significant variables.

Regression Model:

$y = a_o + a_1 (x_2) + a_2 (x_6) + a_3 (x_6^2)$ y = % change

$a_o = -2.2182$

$a_1 = 1.056$

$a_2 = 0.4803$

$a_3 = 3.1425$

Standard Error of Estimate = 1.74%

Analysis of Variance:

Source	Sum of Squares	Degrees of Freedom	Mean Squares	F-Value	% Contribution
x_2	29.34	1	29.34	9.65	21.93
$x_6+x_6^2$	43.72	2	21.86	7.19	32.69
Residual	60.70	20	3.04		45.38
Total	133.76	23			100.00

Standard $F_{1,20}$ = 4.35 @ 0.05 probability point.

Standard $F_{2,20}$ = 3.49 @ 0.05 probability point.

SRL 1317 13 0674

TABLE 4-7 ANALYSIS ON STABILITY OF
CARBON MONOXIDE AT 100 PPM

Multiple Stepwise Regression:

Only cylinder type (x_2) had any significant effect.

Regression Model:

$y = a_o + a_1(x_2)$ y = % change

$a_o = -1.4924$

$a_1 = 1.2260$

Standard Error of Estimate = 2.12%

Analysis of Variance:

Source	Sum of Squares	Degrees of Freedom	Mean Squares	F-Value	% Contribution
x_2	36.07	1	36.07	8.02	26.72
Residual	98.90	22	4.50		73.28
Total	134.97	23			100.00

Standard $F_{1,22} = 4.30$ @ 0.05 probability point.

SRL 1317 13 0674

TABLE 4-8 ANALYSIS ON STABILITY OF
CARBON MONOXIDE AT 500 PPM

Multiple Stepwise Regression:

No variables have any significant effect.

Regression Model:

$y = a_o$ y - % change

$a_o = 0.5444$

Standard Error of Estimate = 1.74%

The percent change in concentration for all 24 cylinders is small enough to be attributable to experimental errors alone. The % change in concentration is independent of the variables considered.

SRL 1317 13 0674

TABLE 4-9 ANALYSIS ON STABILITY OF
CARBON MONOXIDE AT 1000 PPM

Multiple Stepwise Regression:

No variables have any significant effect.

Regression Model:

$y = a_0$ y = % change

$a_0 = 0.1580$

Standard Error of Estimate = 1.06%

The percent change in concentration for all 24 cylinders is small enough to be attributable to experimental errors alone. The percent change in concentration is independent of the variables considered.

SRL 1317 13 0674

TABLE 4-10 ANALYSIS ON STABILITY OF PROPANE AT 3 PPM

Multiple Stepwise Regression:

Only valve type (x_3) is of any significance.*

Regression Model:

$y = a_o + a_1 (x_3)$ y = % change

$a_o = 0.1225$

$a_1 = 0.5076$

Standard Error of Estimate = 1.11%

Analysis of Variance:

Source	Sum of Squares	Degrees of Freedom	Mean Squares	F-Value	% Contribution
x_3	6.18	1	6.18	4.98	18.5
Residual	27.25	22	1.24		81.5
Total	33.43	23			100.00

Standard $F_{1,22} = 4.30$ @ 0.05 probability point.

*Note: x_3 is just barely significant. Moreover, the percent change in concentration for all 24 cylinders is well within experimental errors and no real significance can be attached to any cause-effect relation between valve type and propane stability in cylinders at the 3 ppm level.

SRL 1317 13 0674

TABLE 4-11 ANALYSIS ON STABILITY OF PROPANE AT 10 PPM

Multiple Stepwise Regression:

No variables have any significant effect.

Regression Model:

$y = a_o$ y - % change

$a_o = 0.6797$

Standard Error of Estimate = 0.59%

The percent change in concentration for all 24 cylinders is small enough to be attributable to experimental errors alone. The percent change in concentration is independent of the variables considered.

SRL 1317 13 0674

TABLE 4-12 ANALYSIS ON STABILITY OF PROPANE AT 50 PPM

Multiple Stepwise Regression:

No variables have any significant effect.

Regression Model:

$y = a_o$ $\qquad$ y = % change

$a_o = 0.7477$

Standard Error of Estimate = 0.69%

The percent change in concentration for all 24 cylinders is small enough to be attributable to experimental errors alone. The percent change in concentration is independent of the variables considered.

SRL 1317 13 0674

TABLE 4-13 ANALYSIS ON STABILITY OF PROPANE AT 100 PPM

Multiple Stepwise Regression:

Only cylinder type (x_2) is of any significance.*

Regression Model:

$y = a_o + a_1 (x_2)$ $\quad$ y = % change

$a_o = -0.6262$

$a_1 = -0.3229$

Standard Error of Estimate = 0.59%

Analysis of Variance:

Source	Sum of Squares	Degrees of Freedom	Mean Squares	F-Value	% Contribution
x_2	2.50	1	2.50	7.20	24.60
Residual	7.65	22	0.35		75.40
Total	10.15	23			100.00

Standard $F_{1,22} = 4.30$ @ 0.05 probability point.

*Note: The effect of cylinder type, although statistically significant, does not have any practical significance. The sum of squares contribution due to x_2 is minimal in this case and the F-value appears significant because the residual term is very small in comparison.

SRL 1317 13 0674

TABLE 4-14 ANALYSIS ON STABILITY OF PROPANE AT 500 PPM

Multiple Stepwise Regression:

No variables have any significant effect.

Regression Model:

$y = a_o$ $\qquad$ y = % change

$a_o = 0.3791$

Standard Error of Estimate = 0.38%

The percent change in concentration for all 24 cylinders is small enough to be attributable to experimental errors alone. The percent change in concentration is independent of the variables considered.

The standard error of estimate term shown in these tables gives an estimate of the variance about regression and represents the error with which any response in the dependent variable could be predicted from the independent variables. Note that the square of the standard error of estimate is the residual term. The residual term, as described earlier, is the estimate of the variance which deviates from regression. The least squares property minimizes this sum of squares of residuals in order to estimate the regression coefficients. Residuals are therefore a form of error which cannot be explained in terms of the independent variables used in the model. The F-values shown in the tables are the ratio of the mean squares due to a "significant" independent variable and the mean squares due to the residual variance. Standard F-values are presented below the analysis of variance tables.

4.3 SUMMARY OF EFFECTS OF TEST VARIABLES ON GAS STABILITY

The results of the analysis indicate that the cylinder type has a definite influence on the stability of carbon monoxide at the lower and middle concentration levels (10 ppm, 50 ppm and 100 ppm). Approximately 25% of the variation in the deterioration of carbon monoxide at these levels is explainable by the type of material used in cylinder construction. Carbon monoxide mixtures in cylinders of manganese steel were more stable than those stored in cylinders of chrome-moly steel. These conclusions were reenforced by observation of rust in all the chrome-moly cylinders, as discussed further in Section 5.0.

The effect of diluent purity could not be conclusively established, although it appears that at the lower carbon monoxide concentration levels (10 ppm and 50 ppm) there might be some degree of influence. No interaction effects between cylinder type and diluent purity could be determined.

At the middle and higher concentration levels (especially 500 ppm and 1000 ppm), the percent change in concentration is small enough to be well within the analytical errors of concentration analysis, and none of the variables considered had any dominating effect. Valve material, type of mixing and preconditioning had a negligible effect on the stability of the test carbon monoxide cylinders.

SRL 1317 13 0674

Analysis on the stability of propane in high pressure cylinders indicated that at all levels of concentrations, the deterioration over a six month period was insignificant. The maximum percent change in concentration for a single cylinder was 2.7% at the 3 ppm level, which is within the ±3% specification established as a criteria for stability.

4.4 EFFECT OF EXTREME STORAGE TEMPERATURES ON GAS STABILITY

Changes in the temperature of the environment in which the gas mixtures are stored can effect the equilibrium between the gas phase and the cylinder walls. This shift in equilibrium can be determined from measured changes in concentration before and after the temperature conditioning.

For the purposes of this study, 16 of the 24 cylinders at each level were placed in hot and cold temperature storage. The remaining eight cylinders were designated as control cylinders and not subjected to any extreme ambient influences. Each of the 16 test cylinders were stored for two 2-week periods at -10 to +20°F and at 90 to 100°F. Thus at each level 32 pairs of before-after concentration data were obtained for a "cold-cycle" and a similar set of 32 paired observations determined for the "hot cycle."

A well known test for comparing "treatment" effects on paired data is to perform a t-test on the differences of the pair observations. By taking differences, extraneous effects which might influence both members of a pair tend to cancel out, thus leaving only the effect (if any) of the "treatment."

Table 4-15 is an example of the t-test performed for "hot-cycle" effects on propane mixtures at the 100 ppm nominal concentration level. A listing of the paired differences in ascending order of magnitude is presented, followed by an illustration of the frequency distribution divided arbitrarily into ten groups. The terms are defined as follows:

SRL 1317 13 0674

TABLE 4-15 t-TEST ON HOT-CYCLE EFFECT FOR PROPANE
AT 100 PPM NOMINAL CONCENTRATION

-1.5100
-1.3000
-1.2600
-.6900
-.6200
-.6100
-.5100
-.4900
-.4400
-.3800
-.3500
-.3700
-.3100
-.2600
-.2400
-.2300
-.1900
-.1100
-.0900
.0900
.1000
.1400
.2000
.3500
.4100
.6900
1.3100
1.1700
1.2600
1.2000

3.0 .0 1.0 6.0 0.0 5.0 3.0 1.0 1.0 3.0

-1.5100 - 1.2000
-.2200
-.1134
.69222
.13236
-0.93

SRL 1317 13 0674

x_i: Paired differences

R: Range = x_{max} & x_{min}

Md: Median observation

M_n: Mean = $\frac{\sum_{i}^{n} x_i}{n} = \bar{x}$

SD = Standard Deviation = $\sqrt{\frac{\sum_{i}^{n} (x_i - \bar{x})^2}{n-1}}$

SE = Standard Error = $\frac{SD}{\sqrt{n}}$

t = Computed t-value = $\frac{\bar{x}}{SD/\sqrt{n}} = \frac{\bar{x}}{SE}$

The test is now carried out by considering the hypothesis, H: $\mu_1 = \mu_2$. That is, the "before" and "after" treatment observations came from a universal population with equal means. In other words, there is no effect of treatment on the two sets of observations. The assertion of the hypothesis is stated with some degree of risk. This is termed the level of significance (α) and for this case an α of 0.05 is chosen. From standard t-tables, for an 0.05 level of significance and 31 degrees of freedom, the critical t region is $-2.04<t<2.04$. Therefore the hypothesis is rejected if $t<-2.04$ or $t>2.04$. Since the computed t = -0.93 the hypothesis is accepted that hot temperature storage had no effect on propane at 100 ppm (with a 5% risk of being wrong).

A summary of the effects of hot and cold storage on propane stability is given in Table 4-16. Similar data for carbon monoxide are given in Table 4-17. Only two t values were not between -2.04 and 2.04, and in both cases the average change was 0.1 ppm or less. On this basis it is concluded that hot or cold storage had no significant effect on either propane or carbon monoxide stability at any concentration level.

SRL 1317 13 0674

TABLE 4-16 SUMMARY OF HOT AND COLD STORAGE EFFECTS ON PROPANE STABILITY

Hot/Cold	Conc. (ppm)	Mean Difference (ppm)	Standard Dev.	t Value
Hot	500	0.124	3.210	0.22
Hot	100	-0.113	0.692	-0.93
Hot	50	0.006	0.363	0.09
Hot	10	0.043	0.082	2.92
Hot	3	-0.010	0.053	-1.03
Cold	500	0.653	2.646	1.40
Cold	100	-0.005	0.572	-0.14
Cold	50	0.058	0.474	0.69
Cold	10	0.005	0.098	0.31
Cold	3	-0.143	0.047	-1.73

TABLE 4-17 SUMMARY OF HOT AND COLD STORAGE EFFECTS ON CARBON MONOXIDE STABILITY

Hot/Cold	Conc. (ppm)	Mean Difference (ppm)	Standard Dev.	t Value
Hot	1000	-3.465	20.344	-0.96
Hot	500	1.408	7.322	1.09
Hot	100	-0.587	1.686	-1.97
Hot	50	-0.239	0.727	-1.86
Hot	10	-0.051	0.384	-0.75
Cold	1000	3.183	19.547	0.92
Cold	500	1.327	8.209	0.91
Cold	100	0.086	1.686	0.29
Cold	50	0.193	1.044	1.04
Cold	10	0.116	0.235	2.79

SRL 1317 13 0674

4.5 EFFECT OF CYLINDER PRESSURE ON GAS STABILITY

At each concentration level of carbon monoxide or propane the pressure in 16 cylinders was periodically reduced from a blending pressure of about 2000 psi to about 500 psi at the end of six months. The eight control cylinders were maintained at essentially the blending pressure.

To determine the effect of reduction in cylinder pressure on the stability of the trace gases, the percent change in concentration of the controlled and uncontrolled cylinders over a six month period was subjected to a t-test. The t-test here is on the means of two populations with unequal variances. The hypothesis states that the two populations have the same mean, or in other words, there is no effect of reduction in cylinder pressure on the stability of trace gases. A 0.05 level of significance is chosen and the t-statistic is computed as:

$$t = \frac{(\bar{x}_1 - \bar{x}_2)}{(S_1^2/N_1) + (S_2^2/N_2)} \quad ,$$

and the degrees of freedom are computed as:

$$df = \frac{(\frac{S_1^2}{N_1} + \frac{S_2^2}{N_2})^2}{\frac{(\frac{S_1^2}{N_1})^2}{N_1 + 1} + \frac{(\frac{S_2^2}{N_2})^2}{N_2 + 1}} - 2$$

where:
$\bar{x}_1$ = mean of concentration changes from control cylinders.
$\bar{x}_2$ = mean of concentration changes from uncontrolled cylinders.
S_1^2 = variance of concentration changes from control cylinders.
S_2^2 = variance of concentration changes from uncontrolled cylinders.

From the computed degrees of freedom and for a 0.05 level of significance, the critical t region is obtained at each concentration level of carbon monoxide and propane. If the computed t value lies outside this critical region, the hypothesis that reduction in cylinder pressure has no effect on the stability of trace gases in cylinders is rejected.

Table 4-18 presents the results of this t-test for carbon monoxide and propane. All the computed t values lie well within the critical region and hence the hypothesis that cylinder pressure has no effect on the stability of trace gases in cylinders is accepted.

4.6 EVALUATION OF THE EFFECT OF REGULATOR TYPE

A separate test program was performed to study the effect of cylinder regulator type on the concentration of carbon monoxide and propane in the test mixtures. In this study three control cylinders of carbon monoxide in nitrogen and five control cylinders of propane in air were used. The regulator types evaluated were:

1. Scott Model 2A - single stage, forged brass body, neoprene diaphragm.
2. Scott Model 10A - two stage, forged brass body, Teflon-faced diaphragm.
3. Scott Model 12A - single stage, nickel plated brass body, stainless steel diaphragm, Kel-F and nylon seal.

In addition to the three regulators, a nickel plated brass needle valve was used without a regulator. Past experience had shown that the needle valve, while not practical for general use, eliminated trace gas adsorption and was particularly effective for transferring low concentrations of high molecular weight ($>C_6$) hydrocarbons.

Each test cylinder was analyzed using each regulator and the needle valve in three separate runs. The instrument response for each regulator was then compared to that obtained with the needle valve. The data for carbon monoxide are shown in Table 4-19. The data show that the regulators delivered well in excess of 99% of the concentration delivered by the needle valve except for the 98.6% delivery found for the Model 10A at the two higher concentrations. The slightly lower concentration found

TABLE 4-18 EFFECT OF REDUCED CYLINDER PRESSURE ON STABILITY OF TRACE GASES

Component	Conc. (ppm)	$\bar{x}_1$	$\bar{x}_2$	S_1^2	S_2^2	df	Computed t	Critical Region
Carbon Monoxide	10	-3.996	-5.869	66.484	180.551	23	0.42	$-2.07<t<2.07$
	50	-0.532	0.294	6.151	5.804	15	-0.78	$-2.13<t<2.13$
	100	-0.547	-1.965	2.534	7.100	23	1.63	$-2.07<t<2.07$
	500	0.367	0.633	1.453	3.716	23	-0.41	$-2.07<t<2.07$
	1000	-0.120	0.297	0.978	1.189	17	-0.94	$-2.11<t<2.11$
Propane	3	0.658	0.145	1.577	1.264	14	0.98	$-2.15<t<2.15$
	10	0.445	0.797	0.316	0.335	16	-1.43	$-2.12<t<2.12$
	50	0.609	0.817	1.109	0.197	9	-0.54	$-2.26<t<2.26$
	100	-0.892	-0.493	0.202	0.526	23	-1.66	$-2.07<t<2.07$
	500	0.481	0.362	0.619	0.157	9	0.40	$-2.26<t<2.26$

$\bar{x}_1$ = mean % change in control cylinders.

$\bar{x}_2$ = mean % change in reduced pressure cylinders.

S_1^2 = variance of change in control cylinders.

S_2^2 = variance of change in reduced pressure cylinders.

df = degrees of freedom.

TABLE 4-19 COMPARISON OF REGULATOR TYPES IN ANALYSIS OF CARBON MONOXIDE

Cylinder No.	Conc. (ppm)	Run #	Instrument Response (Cm) for Model 2A	10A	12A	N.V.	Ave. Response as % of N.V. 2A	10A	12A
A-11403	1105	1	19.83	19.43	19.66	19.97			
		2	19.83	19.67	19.99	19.78			
		3	19.02	19.08	19.28	19.22			
		Ave.	19.56	19.39	19.64	19.66	99.5	98.6	100.0
A-11481	110	1	15.49	15.30	15.53	15.56			
		2	15.47	15.39	15.53	15.56			
		3	15.03	14.77	14.99	15.05			
		4	15.17	15.10	15.00	15.27			
		Ave.	15.29	15.14	15.26	15.36	99.5	98.6	99.3
A-11539	10.3	1	6.92	6.96	6.96	7.00			
		2	6.94	6.92	6.93	6.96			
		3	7.03	7.03	7.01	7.05			
		Ave.	6.96	6.97	6.97	7.00	99.4	99.6	99.6

N.V. - needle valve.

with the Model 10A (two stage) was reasonably consistent from run to run, but it is within experimental error for the analyzer.

The evaluation of regulators for use with propane in air mixtures included measurements of total hydrocarbons as well as propane because some regulators, especially those with rubber diaphragms, have been known to add hydrocarbons to the gas passing through them. The propane tests were conducted similarly to the carbon monoxide tests described above. The data are given in Table 4-20. Two of the test cylinders (A-11526 and A-11534) were blended with hydrocarbon-free synthetic air, and thus the propane and total hydrocarbons are similar. The other three cylinders were blended with synthetic air containing methane and other hydrocarbons (see page 2-5). In these cases the total hydrocarbons exceed the propane.

The data for both propane and total hydrocarbons show variations well within the experimental error of the instruments. This indicates that any regulator type tested may be used. However, caution must be exercized. The test regulators were all new and had not been exposed to higher molecular weight hydrocarbons. These compounds can readily adsorb on inside regulator surfaces and be desorbed in subsequent use. Thus, when first using any regulator with uncertain previous use history, it is desirable to test the regulator with hydrocarbon-free air to determine if any previously adsorbed hydrocarbons will be released into the gas stream.

TABLE 4-20 COMPARISON OF REGULATOR TYPES IN ANALYSIS OF PROPANE

Cylinder No.	Propane Conc. (ppm) for Model				Total HC as Propane (ppm) for Model		
	2A	10A	12A	N.V.	2A	10A	12A
A-11498	514.0	515.0	515.3	513.6	527.5	528.0	529.8
A-11487	106.2	106.1	106.5	106.3	109.5	109.0	109.4
A-11526	53.3	53.3	53.4	53.2	52.7	53.1	52.8
A-11505	17.42	17.35	17.40	17.38	19.60	19.84	19.81
A-11534	3.04	3.03	3.01	3.05	3.02	3.07	3.02

N.V. - needle valve

5.0 DISCUSSION OF RESULTS

5.1 STABILITY OF CARBON MONOXIDE MIXTURES

The most significant finding of this study was the substantial losses in carbon monoxide concentration which occurred when mixtures were prepared in chrome-moly cylinders. This was most evident at the low concentration levels (10 to 100 ppm). This loss of carbon monoxide was not entirely unexpected since similar poor stability had been experienced in actual use conditions in random cylinders. However, no study had been made on a sufficiently large batch of cylinders to pinpoint the cause of instability.

The interior surfaces of those cylinders in which significant losses occurred were examined and compared to those with good stability. The surfaces of the manganese steel cylinders which provided good stability were reasonably smooth and coated with a gray-black film. On the other hand, troublesome chrome-moly cylinders had surfaces with large rust colored blotches. Those chrome-moly cylinders in which the greatest losses of carbon monoxide occurred appeared to have more rust colored areas than those which exhibited modest loss in carbon monoxide. It can be theorized that the rusty surfaces lead to carbon monoxide losses by accelerating the formation of iron carbonyls. No efforts were made to pursue this theory because the extensive work necessary could not be justified in terms of its practical value.

The conclusion that the use of chrome-moly cylinders for storage of 10 to 100 ppm carbon monoxide mixtures is likely to produce poor stability is clearly demonstrated by the data in Table 4-2. Thus, it is recommended that manganese steel cylinders be used for these mixtures until it can be demonstrated that other materials provide equivalent stability. The only disadvantage of manganese steel cylinders is their additional weight of 20 pounds which leads to higher transportation costs. The additional costs are very modest when compared to the value of a stable calibration gas mixture.

Some chrome-moly cylinders had much greater losses than others at the same concentration level. This random effect tended to mask the influence of the other test variables. Diluent purity produced a statistically significant effect at the 50 ppm level. The regression analysis indicates that the diluent nitrogen with lower purity (higher oxygen content) tended to accelerate losses of carbon monoxide. Where the diluent also contained higher moisture, this effect appears to have been reversed. The use of high purity nitrogen (>99.997%) in manganese cylinders produced satisfactory stability at all concentration levels. It is readily available at low cost and thus should be the diluent of choice for carbon monoxide mixtures, especially in the 10 to 100 ppm range.

The other test variables: Valve type, cylinder preparation procedure and mixing procedure had no effect at any concentration level. Any of the valves or preparation and mixing procedures used should be satisfactory for producing stable mixtures of carbon monoxide in nitrogen.

5.2 STABILITY OF PROPANE MIXTURES

All of the 120 test cylinders of propane in air satisfied the stability criteria of less than 3% change in six months. The only variables shown to have a statistically significant effect at the 95% confidence level were valve type at 3 ppm and cylinder type at 100 ppm. In both cases the effect was less than 1%, thus within experimental error of the analytical measurements.

While the purity of the diluent gas had no effect on the stability of the propane, it should be pointed out that the hydrocarbons other than propane present in the diluent can lead to serious calibration errors when the propane mixture is used for calibration of flame ionization detector total hydrocarbon analyzers. It is generally assumed by the user that the propane represents the total hydrocarbons in the mixture. The test cylinders prepared with blended air and water pumped air contained

approximately 1.8 and 1.0 ppm propane equivalent respectively, of other hydrocarbons. Errors due to non-propane hydrocarbons can be reduced by having the cylinder analyzed for total hydrocarbon by the supplier. For best results propane mixtures should be prepared in hydrocarbon-free air (<0.1 ppm-C) and analyzed for both propane and total hydrocarbons. This procedure will detect cases where hydrocarbons may have entered the cylinders from improperly flushed manifolds or other sources.

5.3 STORAGE OF CARBON MONOXIDE AND PROPANE MIXTURES

The data analysis described in Section 4.4 showed that storage at 0^{o} and 95^{o} F had no measurable effect on stability of carbon monoxide or propane mixtures in the range of concentrations tested. Low temperature storage of higher molecular weight hydrocarbons can lead to losses through condensation on cylinder walls. This had been a serious problem when hexane was used as the standard hydrocarbon. High temperature storage can accelerate reactions which cause losses of mixture components. It can also result in desorption of material present from previous cylinder useage.

This study has shown that mixtures of carbon monoxide and propane can be stored over a wide temperature range without loss of stability. It is nevertheless desirable to allow the cylinders to equilibrate to room temperature before use. In any event, the temperature of the calibration gas and sample gas to be analyzed must be at the same temperature if an instrument is to produce accurate data.

5.4 GENERAL COMMENTS

This study has been limited to effects where new cylinders, valves and regulators are used. Any attempt to evaluate situations where materials had been exposed to other mixtures would have required a program of impractically large magnitude. In actual practice, cylinders are refilled many times in their useful life. Most suppliers try to re-use a cylinder for the same type of mixture as it previously contained. Periodic cleaning of cylinders may be performed.

This does not mean that cylinders should not be refilled or even that refilled cylinders are more likely to lead to more stability problems than new cylinders. Rather, it must be realized that producing gas mixtures following the recommendations developed in this program will assure but not guarantee satisfactory stability. The possibility always exists that an unknown and indeterminable factor will reduce stability. Thus, in addition to purchasing gases prepared to the recommended specifications, a user should plan a periodic check of his calibration mixtures. This is best accomplished by participation in a commercial gas cross-reference service, but comparison with primary standards or other mixtures in possession of the user can also serve to check stability.

Inaccurate span and zero gases are most probably the largest single contributors to errors in gas concentration measurements. Therefore, quality assurance in the manufacture and use of gas mixtures is essential to all programs involving air monitoring or measurement of emissions from mobile and stationary sources.

6.0 RECOMMENDATIONS

6.1 CARBON MONOXIDE IN NITROGEN MIXTURES

Based on the data analysis and other relevant information acquired in this study, the following practices are recommended for producing stable mixtures of carbon monoxide in nitrogen in the range of 10 to 1000 ppm:

1. Mixtures of 10 to 500 ppm must be blended in manganese steel cylinders meeting DOT Specification 3A2015. While the use of chrome-moly cylinders is satisfactory at concentrations above 500 ppm, substantial losses frequently occur in the 10-100 ppm range.
2. Cylinder valves may be either brass with Teflon packing or diaphragm packless brass.
3. For new cylinders, preconditioning by evacuation at ambient temperature to 0.1 in. Hg absolute pressure is adequate.
4. Mixing after blending may be accomplished by either mechanical or thermal methods.
5. The diluent nitrogen should have a purity of 99.997% or greater. The use of diluent gas with a lower purity (higher oxygen content) can accelerate loss of carbon monoxide. This effect may cause unsatisfactory stability in the 10 to 100 ppm carbon monoxide range.

The following recommendations apply to the storage and use of carbon monoxide in nitrogen mixtures:

1. Cylinders may be stored at temperatures from -10 to 100°F without loss of stability. For best results, the cylinders should be stored in the laboratory for 24 hours so that the contents reach ambient temperature before use.
2. Cylinder regulators having bodies of brass, aluminum or stainless steel and diaphragms of neoprene, Teflon-faced metal or stainless steel are all satisfactory.

SRL 1317 13 0674

6.2 PROPANE IN AIR MIXTURES

Based on the data analysis and other relevant information acquired in this study, the following practices are recommended for producing stable mixtures of propane in air from 3 to 500 ppm:

1. Mixtures may be blended in steel cylinders meeting DOT Specification 3AA2015 or 3A2015.
2. Cylinder valves may be either brass with Teflon packing or diaphragm packless brass.
3. For new cylinders, preconditioning by evacuation at ambient temperature to 0.1 in. Hg absolute pressure is adequate.
4. Mixing after blending may be accomplished by either mechanical or thermal methods.
5. Satisfactory propane stability can be achieved using blended hydrocarbon-free air, blended nitrogen and oxygen or water pumped air as the diluent gas. However, the use of hydrocarbon-free air offers the advantage of excluding methane and other hydrocarbons which must be taken into account when using the gas mixtures for calibration of flame ionization total hydrocarbon analyzers.
6. All cylinders should be analyzed for total hydrocarbons as well as propane. This will determine whether other hydrocarbons have entered the mixture via the diluent gas, dirty blending system, cylinder residuals, etc.

The following recommendations apply to the storage and use of propane in air mixtures:

1. Cylinders may be stored at temperatures from -10 to 100°F without loss of stability. For best results, the cylinders should be stored in the laboratory for 24 hours so that the contents reach ambient temperature before use.

2. Cylinder regulators having bodies of brass, aluminum or stainless steel and diaphragms of neoprene, Teflon-faced metal or stainless steel may all be used if clean and new. Past experience has shown that neoprene diaphragms can cause problems if they have been exposed to higher hydrocarbons. Thus, extreme caution must be exercised with neoprene diaphragms, and it is a better practice to use metal or Teflon-faced diaphragms.

7.0 ACKNOWLEDGEMENTS

We wish to express our thanks to the Project Officer, Mr. John H. Margeson and to Dr. John B. Clements, Chief of the Methods Standardization Branch, Quality Assurance and Environmental Monitoring Laboratory for their assistance in the planning and performance of the gas stability program. Mr. Margeson's comments and helpful suggestions were of great benefit throughout the entire program period.

8.0 REFERENCES

1. Davies, O.L., Goldsmith, P.L., "Statistical Methods in Research and Production," Hafner Publishing Co., New York, 1972.

2. Dixon, N.J., Massey, F.J., "Introduction to Statistical Analysis," McGraw-Hill Book Company, Inc., 1957.

3. Stepwise Multiple Regression program obtained from UNIVAC STAT-PACK Routine: RESTEM.